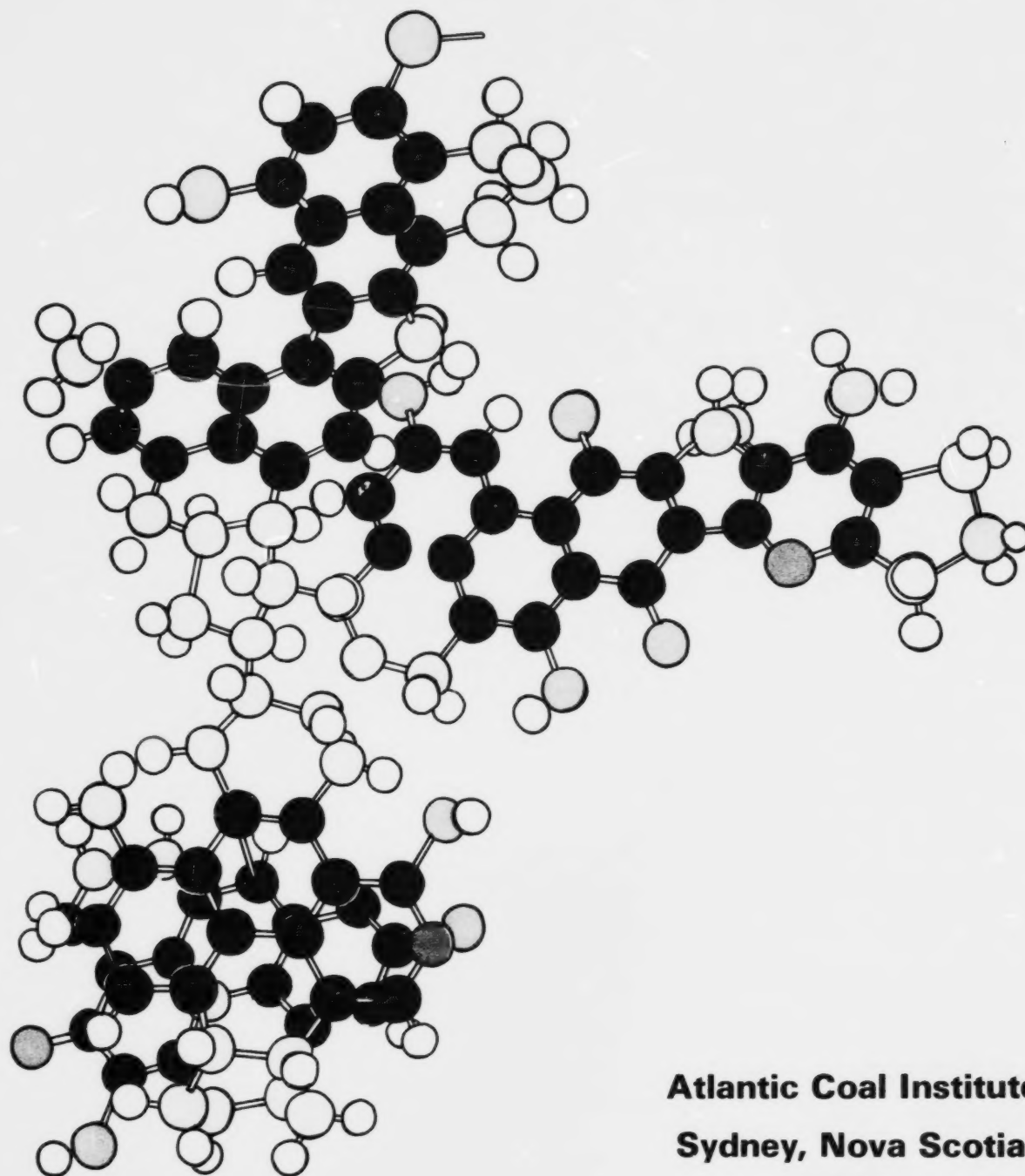


Assessment of Nova Scotia Coal Preparation Problems



**Atlantic Coal Institute
Sydney, Nova Scotia
1982**

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ASSESSMENT OF NOVA SCOTIA COAL PREPARATION PROBLEMS (UP-A-207)

FINAL REPORT

BY

ATLANTIC COAL INSTITUTE

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SUMMARY

The Atlantic Coal Institute submitted an unsolicited proposal to the Department of Energy, Mines and Resources and received a contract to assess Nova Scotia's coal preparation problems, with emphasis on sulphur content.

Five channel samples were taken from different locations in Lingan Mine, operated by the Cape Breton Development Corporation. The samples were taken in six inch increments for a total of seventy separate samples. Each sample was screened at 24 mesh and the plus 24 mesh fraction put through a sink-float process at 1.30 and 1.35 specific gravity. All samples were analyzed for total sulphur, pyritic sulphur and organic sulphur.

The overall effect of the of the sink-float process using 1.35 s.g. was to reduce the total sulphur by 20.8%, with a weight recovery of 85%. When floated at 1.30 s.g., the effect was to reduce the total sulphur by 34.3%, however recovery dropped to 74.2%. Moving from 1.35 s.g. to 1.30 s.g., as expected, did not effect the removal of organic sulphur.

Although the sulphur values vary from both pyritic and organic forms, the organic sulphur can be considered relatively constant in comparison to the pyritic sulphur and where total sulphur shows a significant increase, this is due in general to a corresponding increase in pyritic sulphur.

An analysis of variance could not establish significant variation in percentages of sulphur between samples and horizontal distances.

The relationship between the height of the seam and the sulphur types was examined and suggest a pattern of less total sulphur and pyritic sulphur as the height decreases.

The possibility of a linear relationship between total sulphur and total iron was investigated. One of the problems encountered was the presence of high free iron values. These high values show the presence of iron other than pyritic iron and make the prediction of sulphur in terms of iron unreliable.

A section is presented on trace elements in coal and their possible effect on the environment. This assessment is approached from three sources of possible contamination: mining, preparation plant and generating stations. Easy access to the ocean for runoff is a saving factor in Nova Scotia. Concern is not for the immediate or acute toxicity, but the effect of large quantities of elements cumulating in the environment over the long term.

A revision of the work done on sulphur removal by the Cape Breton Development Corporation and various scientific resources, called the Atlantic Group for Research in Industrial Metallurgy (AGRIM) is included in this report.

The final section of this report deals with Preparation Procedures and concepts applicable to Nova Scotia Coals. The various methods of reducing sulphur and sulphur emissions are discussed under the following:

1. Physical cleaning
2. High gradient magnetic separation
3. Flue gas desulphurization
4. Chemical coal cleaning

5. Gasification of coal
6. Liquification of coal
7. Coal-Oil-Water mixtures
8. Fluidized bed combustion

INTRODUCTION

The present coal reserves are essentially in five areas in the Province of Nova Scotia: Sydney, Port Hood, Mabou, Stellarton, and Springhill. It is estimated that there are three and one-half billion tons of coal in known deposits, of which approximately one billion tons can eventually be mined. The reserves in the Sydney coalfield represent approximately 80 percent of the total.

This project deals with the Sydney coalfield and more specifically with the producing mines and preparation plant operated by the Cape Breton Development Corporation (CBDC).

The energy policy of Nova Scotia suggests that the immediate target should be a production of coal in the range of eight million tons annually or the replacement of about thirty-two million barrels of imported oil per year.

The planned increase in coal production creates the additional problems of coal preparation, not the least of which is the reduction of sulphur to meet environmental considerations.

This study looks at the sulphur content of coal samples taken from the Lingan Mine, in the Sydney coalfield; assessing from the accumulated data questions respecting sulphur and sulphur forms, reviews the work done on sulphur removal to date, and the effect of trace elements in coal.

A review of preparation procedures and concepts which may be applicable to Nova Scotia coals is discussed.

COAL PRODUCTION FROM SYDNEY COALFIELD

The Cape Breton Development Corporation is the largest producer in the area, with three mines presently in operation.

Prince Mine, operating in the Hub Seam will produce about 500,000 tons in 1982 and expand to 1.1 million tons by 1985.

The Lingan Mine operating in the Harbour Seam, will produce 1.5 million tons in 1982 and expand to 1.75 million tons by 1983.

No. 26 Mine, also operating in the Harbour Seam, produces 800,000 tons annually.

Development of Donkin Mine is well underway, expanding from 400,000 tons per year in the mid-1980's to three million tons by the mid-1990's.

Devco is presently assessing the feasibility of a fifth mine, which will produce an additional 1.5 million tons annually.

The location of the Sydney coalfield and other mining areas are shown in Figure 1. The sequence of seams is shown in Figure 2.

Production and expansion figures are from "Coal 2000; Nova Scotia's Perspective" by J.J. Laffin, Deputy Minister of Mines and Energy, presented Quebec, April 26, 1982.¹

COAL PREPARATION REVIEW

The Cape Breton Development Corporation, prior to the construction of the present Victoria Junction Coal Preparation Plant, and not unlike other producers, used washing as a means of stone and refuse removal,

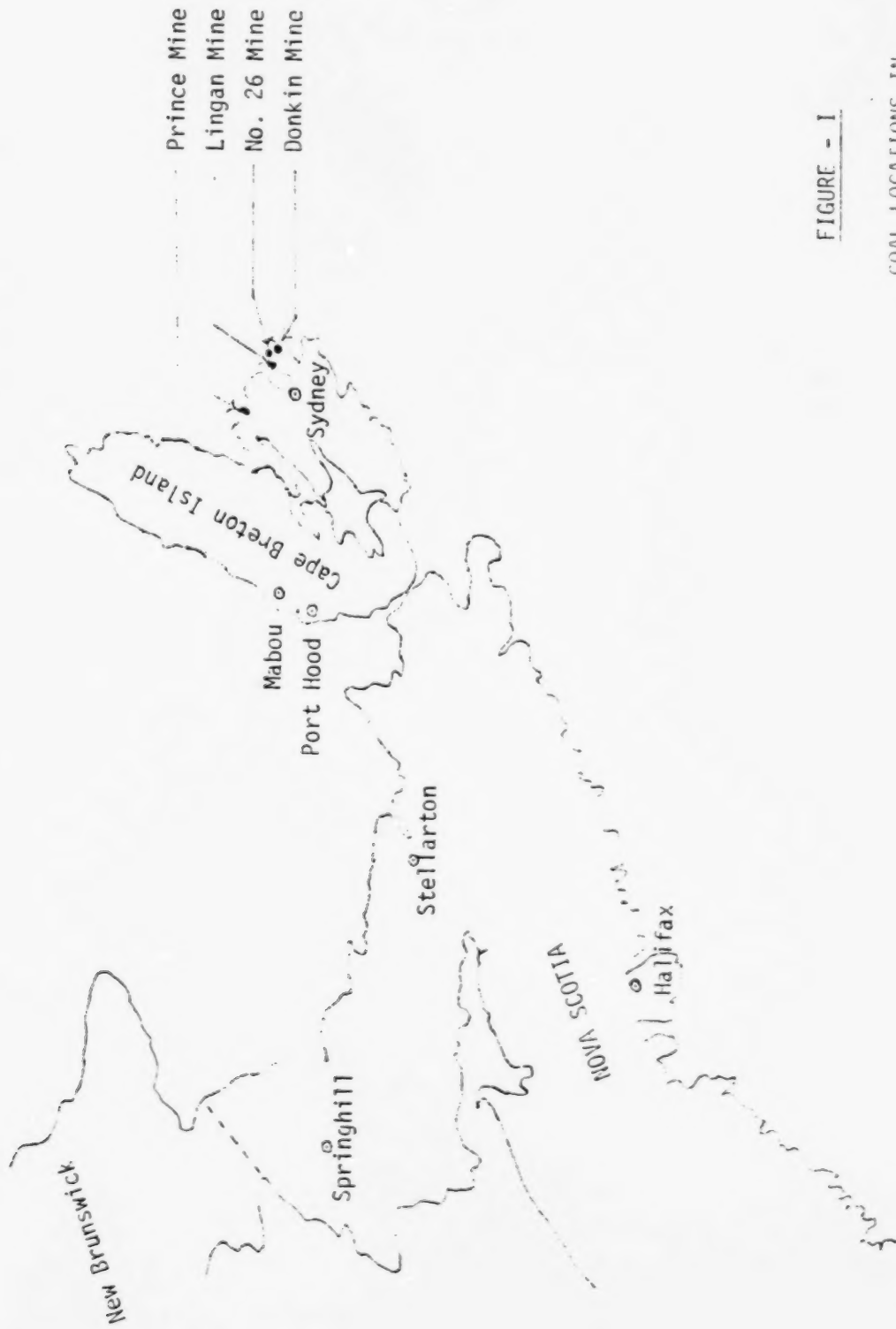
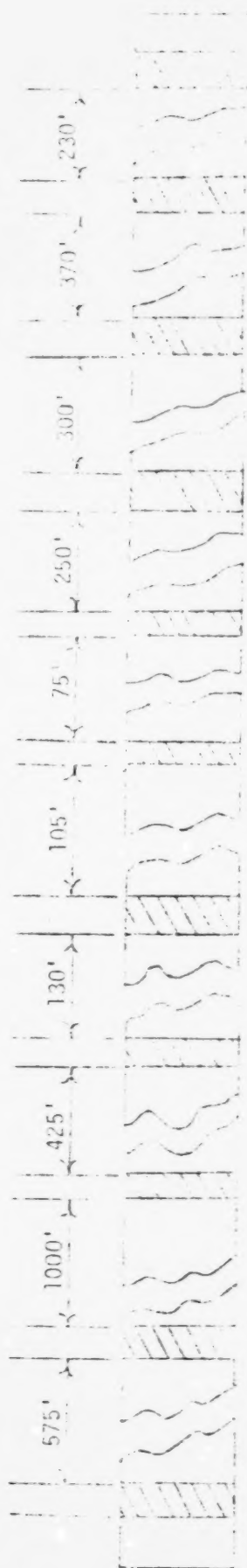


FIGURE - I

COAL LOCATIONS IN
NOVA SCOTIA



Pt. Aconi 3' to 7' Coal

Bonar 4' to 12' Coal (May be split)

Hub Upper 9' Coal
Lower 3' Coal

Harbour 4' to 15' Coal

Bouthillier 3' Coal

Back Pit 3' Coal

Phalen 3' to 8' Coal

Emery 4' Coal

Gardiner 4' Coal

Mullins 4' to 7' Coal

Tracey 4' to 6' Coal

Figure 2

Sydney Coal Field
Sequence of Seams

any sulphur reduction was incidental.

In the late 1960's, No. 26 Colliery was producing coal with a sulphur level low enough that, after washing, it was acceptable in the coking operations of Sydney Steel Corporation.

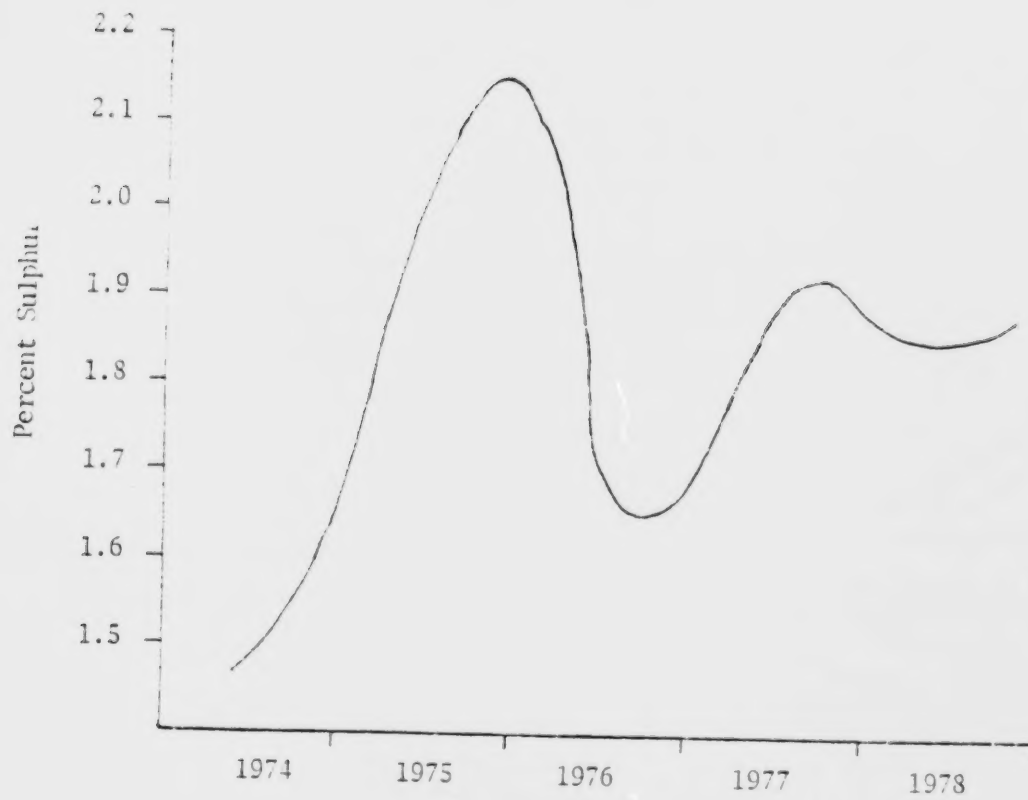
Working closely with the Federal Department of Energy, Mines and Resources, CBDC embarked on a study of both the problem of sulphur removal and possible solutions.

The work of Dr. F.P. Agterberg, using data from No. 26 and No. 12 mines, was able to predict by computer technology, the sulphur content of that block of coal in which the new Lingan Mine would operate. Figure 3, from "Geomathematical Prediction of Sulphur in Coal, New Lingan Mine Area, Sydney Coalfield" by F.P. Agterberg and C.F. Chung,² shows the predicted sulphur from Lingan Longwall operations over the years 1974 to 1978. Agterberg stated that the best estimates they could compute appeared to be rather limited, and in fact the predictions did prove to be lower than the actual sulphur levels encountered.

In 1973, J. Visman and M.W. Mikhail, of CANMET'S Western Research Laboratory, conducted pilot plant studies to confirm that the sulphur content of the coals expected to be produced at Lingan and No. 26 Colliery could be economically reduced.³

It was recognized that the degree of liberation of pyritic sulphur increased as the size of the coal was reduced. However, this crushing would result in a problem of customer acceptance and increase the retained moisture, requiring thermal drying with its economic and environmental problems. An optimum design for a preparation plant was arrived at, producing metallurgical coal to specifications without the need for thermal drying.

From "Agterberg" - Model 1 Coal
Block "A" 3 Walls Operating
Walls starting 5 months apart



Predicted sulphur variation in the Lingan Longwall operation.

FIGURE 3

The new plant processed its first coal in late 1976 and has produced a beneficiated product up to the present.

COAL PREPARATION PLANT (CBDC)

The Coal Preparation Plant is located at Victoria Junction, near Grand Lake, where the rail lines from Lingan Mine and No. 26 Mine meet.

The plant, designed by Simon-Carves of Canada, receives coal from both mines which is brought to the car dumping station by 100 ton capacity rotary-dump rail cars. After unloading at a rate of 2000 tons per hour, the coal is transported via a run-of-mine (R.O.M.) reclaim conveyor and R.O.M. radial stacker to separate stock piles. The coal from each stockpile is transported by its individual reclaim conveyor to the screening and crushing house, where it is screened at 1 1/2 inches, the oversize is crushed and recirculated.

The crushers can be adjusted to produce coal down to 3/4 inches. The underflow from the raw coal screens together with the crushed product, is transported to the launders feeding the Lingan and No. 26 raw coal distributors.

The raw coal feed is flushed by water via the raw coal distributors to the desliming sieve bends, where it is screened at 28 mesh.

The minus 28 mesh undersize, together with the flushing water and the minus 28 mesh undersize from the desliming screens, passes to the froth flotation cells for subsequent treatment.

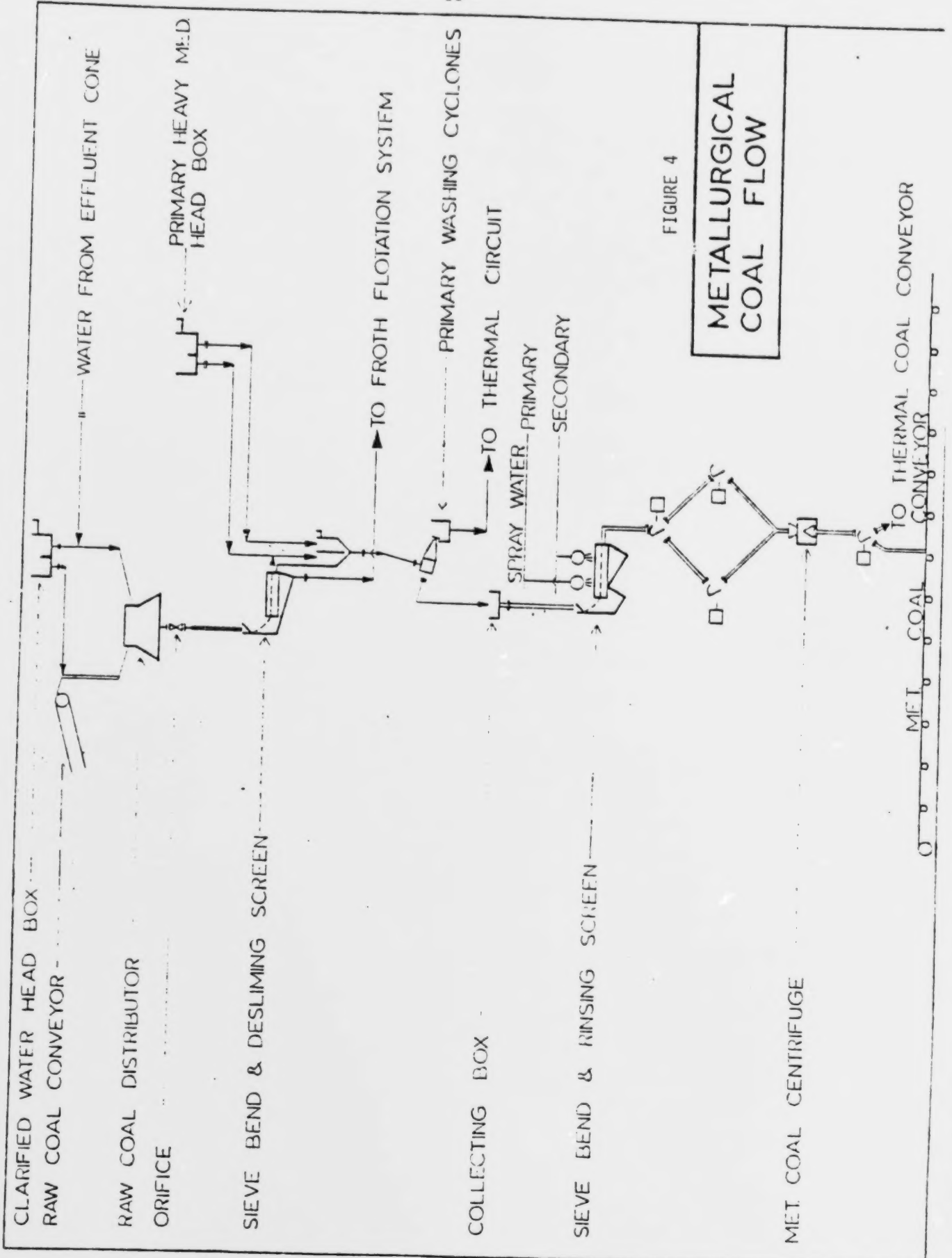
The plus 28 mesh deslimed coal, together with the primary heavy medium, gravitates via the cyclone mixing tanks to the 26 inch diameter washing cyclones. The heavy medium in circulation is a suspension of magnetite and water controlled at predetermined specific gravity.

The cleaned coal, which is now the metallurgical produce, passes to a spraying and draining screen to recover the medium, which is re-circulated. This production of metallurgical coal is based on the expectation that No. 26 coal can be cleaned to 1% sulphur and that Lingan coal can be cleaned to approximately 1.3% sulphur, so that proper mixing of the two coals will attain the desired specification of 1.2% sulphur, or less.

The rejects or sink from both Lingan and No. 26 pass to the secondary heavy medium cone and are pump fed to the secondary 26 inch diameter cyclone for separation at a higher gravity than the primary heavy medium. The overflow or float of the secondary circuit is now the thermal coal, and after medium recovery, passes to the out-loading conveyor. The discard product or sink from the secondary washing cyclones, passes to a wash screen to recover the medium and then via a centrifuge to a discard conveyor.

The minus 28 mesh coal in the flotation cells, plus a measured flow of frothing and collecting agents, is separated into three products, metallurgical, thermal and rejects. The metallurgical and thermal products are collected in their respective launders, and passed via centrifuges to be blended with their cyclone washed products. The rejects from the flotation circuit goes to a 125 foot diameter thickener for clarification. The overflow is recirculated and the tailings are pumped to settling basins.

The metallurgical coal flow is shown in Figure 4, the thermal coal flow in Figure 5 and the fines circuit in Figure 6.



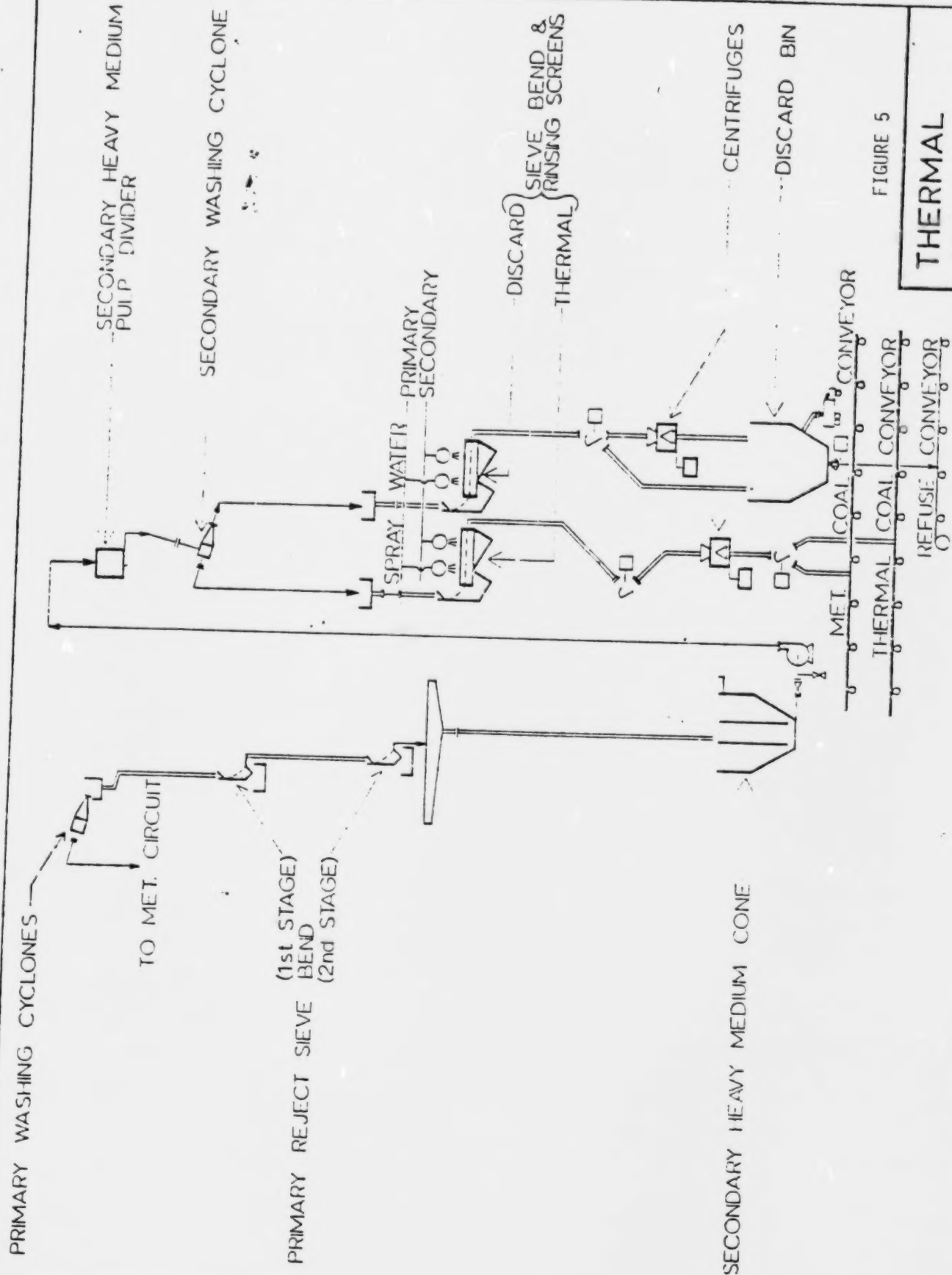


FIGURE 5

THERMAL
COAL FLOW

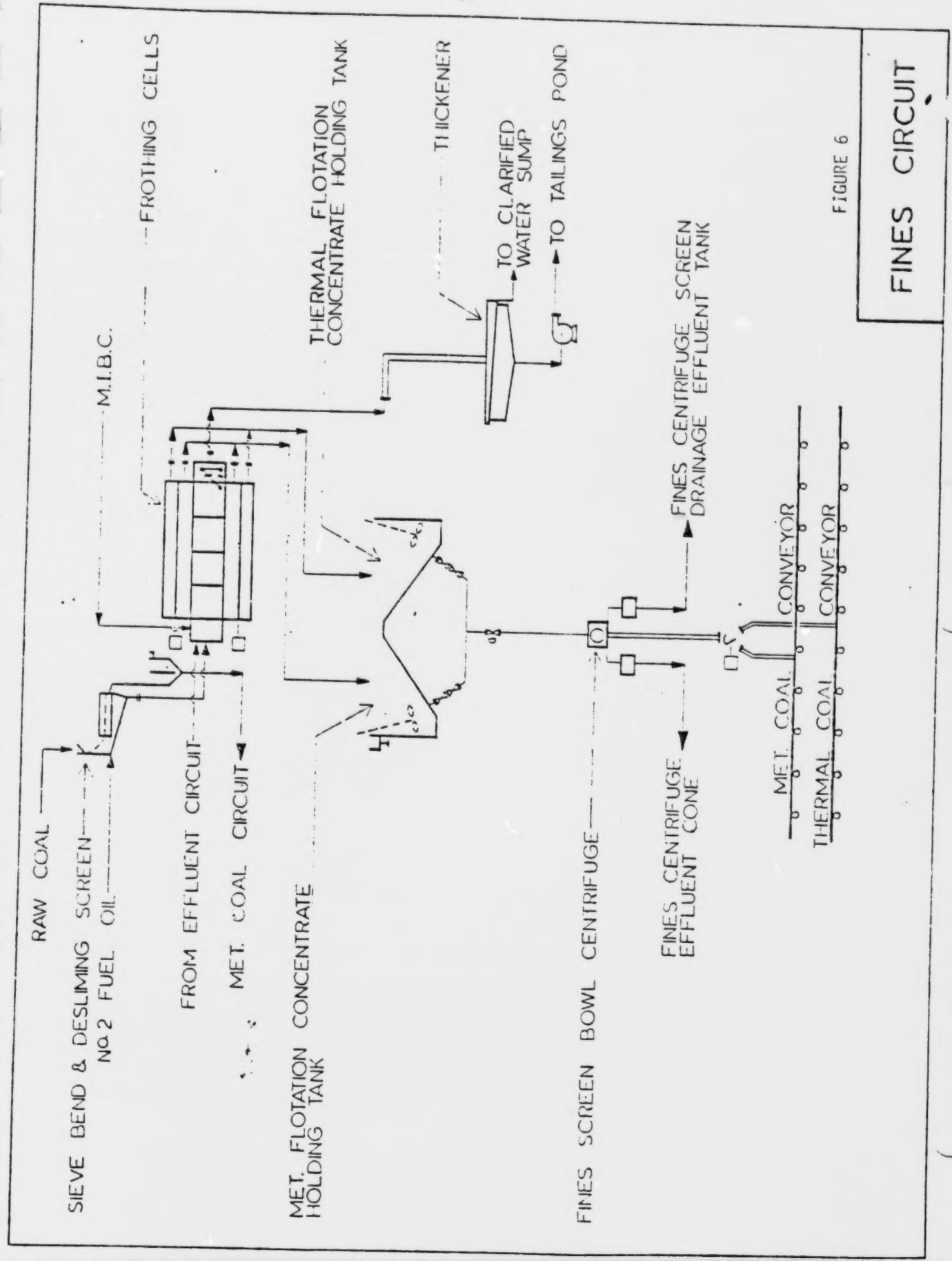


FIGURE 6

FINES CIRCUIT

COAL PREPARATION PROBLEMS

Coal preparation in Nova Scotia, of any great significance, is limited to the Cape Breton Development Corporation's Coal Preparation Plant at Victoria Junction, Cape Breton. The plant is described in detail elsewhere in this report, however, its designed function was to produce a suitable coking coal (sulphur less than 1.2%) while providing thermal coal with higher sulphur values.

The production of metallurgical coal is predicated on the assumption that mixing Lingan and No. 26 coal from the primary washing circuit, will produce coal cleaned to approximately 1.2% sulphur, or less. This is not always the case and depends directly on the sulphur content of the raw feed.

The sulphur content in Lingan mine is higher than that predicted by Dr. Agterbert, and it is difficult and at times impossible to meet specifications for metallurgical coal in quantity.

The rejects or sink from the primary circuit, becomes the thermal coal, and in general this product will also be higher in sulphur when the raw coal is higher. Further adjustments to the specific gravity in this circuit, to remove sulphur, would result in unacceptable losses in the final reject.

The problem with sulphur removal is that there are relative large amounts of organic sulphur which can not be removed with physical cleaning and that much of the pyritic sulphur is fine grained, and difficult to liberate. It is recognized that fine grinding will liberate much of the pyrite, allowing physical cleaning with reduced sulphur. However, this would introduce a new fine coal technology involving flotation

separation and the problem of removing the water. The technology of fine grinding and flotation has sparked the interest in coal-oil-water mixtures which can be burnt in converted oil fired boilers.

In 1976 a federal-provincial task force presented their recommendations to the Environmental Protection Service on emission guidelines for thermal generating stations. These guidelines are still under consideration by E.P.S. and are shown in Table 1.

TABLE 1
PROPOSED NATIONAL EMISSION GUIDELINES
FOR THERMAL GENERATING STATIONS

<u>POLLUTANTS</u>	<u>PROPOSED EMISSIONS (lbs/10⁶BTU)</u>
Participants	0.05 - 0.15
Sulphur Dioxide (SO ₂)	0.5 - 1.60
Nitrogen Oxides	0.70

These guidelines could not be met, without reducing the sulphur in the coal or some method of reducing the SO₂ emissions at the combustion site. Should a standard be set at 1.2 lbs. SO₂ per 10⁶ BTU's (the present U.S.A. standard), then the sulphur content of thermal coal could not exceed 0.81%, assuming a caloritic value of 13,500 BTU/lb. Devco's present thermal coal is in the order of 2.5 - 3.0% sulphur.

The proposed expansion in coal mining and the conversion of Nova Scotia's thermal generating stations from oil to coal will pose difficult and challenging problems in coal preparation. The level of production anticipated will require the construction of at least two new coal preparation plants. The degree of sulphur reduction possible in any new

plant will of necessity be the result of an economic balance, and it would appear that a further method must be found for reducing emissions at the coal fired source.

SULPHUR RESEARCH - LINGAN MINE

It was agreed with CBDC officials that a series of channel samples would be taken in Lingan Mine from the working faces. These samples would be taken over an extended period and the data used to determine possible correlations between sulphur content and distance.

The samples were a series of sectional samples taken at six inch intervals from the roof to the floor of the coal seam. A geological pick was used to extract each interval to a depth of three inches and providing approximately ten pounds of coal for each interval. If a bone band was encountered, it was not taken as a separate section but enclosed with the interval sample.

Each increment was screened at 24 Mesh and the plus 24 Mesh put through a sink float process. The gravity was adjusted using an organic solution trade named "Certigrav," resulting in separations at 1.30 and 1.35 specific gravity. These gravities correspond to the range used in the preparation plant in the primary metallurgical coal circuit.

The various s/f fractions and the minus 24M sample, were pulverized and analyzed for total sulphur. A LECO, SC-32 sulphur determinator was used in this procedure.

Preliminary sulphate analysis was done on several coal samples and all values found were less than 0.05%. This was considered insignificant for our purposes and discontinued. The total sulphur was assumed to be made up of pyritic and organic sulphur.

The sulphur forms, pyritic and organic, were determined by chemical analysis using ASTM D2492-77 as shown in Appendix A. In our procedure, the two separately weighed sample method was used for determining pyritic iron and total iron.

A total of five channel samples were taken in Lingan Mine, two from 7 East Wall, two from 3 West Wall and one from 6 East Wall, as shown in Figure 7.

The five channel samples provided sixty-nine increments and in excess of 800 determinations of total sulphur and sulphur forms. This data is presented in Appendix B.

The total increment analysis was calculated and also appears in the tabulation of data in Appendix B.

Histograms of each channel sample's total increment were prepared illustrating total sulphur, pyritic sulphur and organic sulphur. This graphical data is presented in Appendix B.

DISCUSSION AND RESULTS

The data as presented in Appendix B shows the wide fluctuations of sulphur content within the seam from roof to floor and also between the different sample locations.

Values of total sulphur, within the seam, as in Channel Sample No. 5, vary from 6.91% to 0.54% and again in Channel Sample No. 2, from 3.32% to 0.5%.

The average total sulphur values between channel sample locations ranges from 3.23% in Sample No. 4, to 1.02 in Sample No. 1.

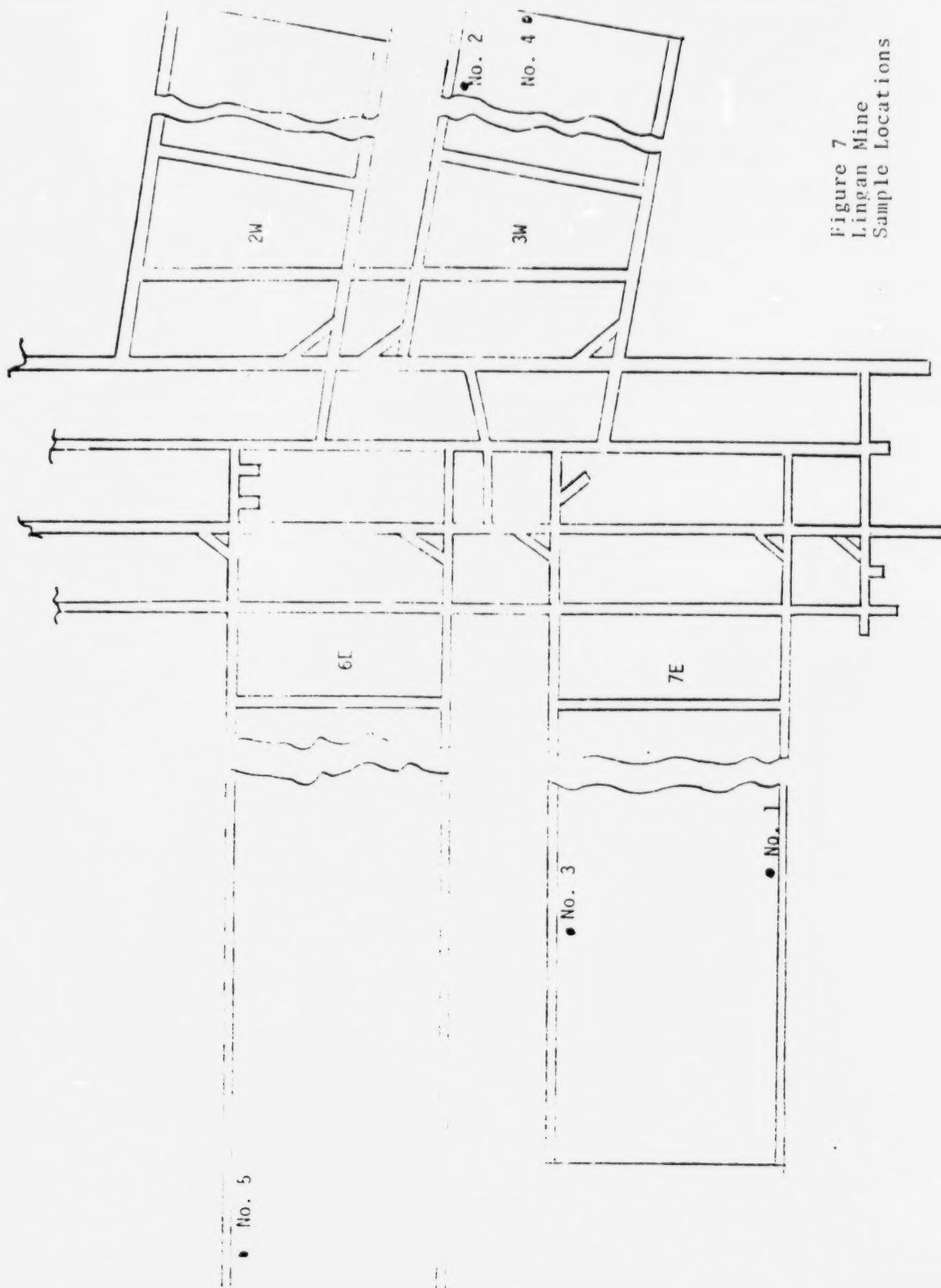


Figure 7
Linggan Mine
Sample Locations

There is a pattern of increased sulphur, in the top and bottom of the seam.

Although the sulphur values vary for both pyritic and organic, the organic sulphur could be considered relatively constant in comparison to the pyritic sulphur and where the total sulphur shows a significant increase this is due in general to a corresponding increase in pyritic sulphur.

A weighted arithmetic average of all samples shows the total sulphur to be 2.07%, the pyritic sulphur 1.45% and organic sulphur 0.62%.

The arithmetic weighted averages of all samples shows, that the effect of floating the coal at 1.35 s.g. resulted in a reduction of total sulphur from 2.07% to 1.64%. The pyritic sulphur was reduced from 1.45% to 0.97%. The organic sulphur indicated a slight increase from 0.62 to 0.67.

When the coal was floated at 1.30 s.g., the total sulphur was reduced from 2.07% to 1.36%. The pyritic sulphur was reduced from 1.45% to 0.69%. The organic sulphur again showed a slight increase, due to the weighted average, from 0.62% to 0.67%.

The overall effect of the s/f process using 1.35 s.g. was to reduce the total sulphur by 20.8% with a weight recovery of 85%. When floated at 1.30 s.g., the effect was to reduce the total sulphur by 34.3%, however recovery dropped to 74.2%.

Moving from a S.G. of 1.35 to 1.30 did not, as expected, effect the removal of organic sulphur. The reduction in sulphur levels at both gravities was due to a reduction in pyritic sulphur.

An attempt was made to establish a relationship between sulphur content and distance between samples, both north-south and east-west.

Such a relationship could become a valuable method of predicting sulphur content in future mining operations.

No significant variations in sulphur content, in relation to distance, could be established, and no pattern emerged. It is possible that sampling was too limited and too localized. All samples came from Lingan Mine at distances east-west 8500 ft., and north-south 2000 ft. It is suggested that this question be the subject of future research and broadened to embrace the whole of the known Sydney coalfield with a systematic sampling program in four directions.

Investigation attempted to answer the question, "can sulphur be related to iron content?" This has a very practical application. The iron content of the coal can be measured directly, and if the relationship with sulphur could be established, then a convenient and economical method of measuring sulphur would be available and provide a valuable method of control in the coal preparation plant.

The weighted iron and sulphur values for the various fractions are shown in Table 2.

TABLE 2

FRACTION	CHANNEL NO. 1		CHANNEL NO. 2		CHANNEL NO. 3		CHANNEL No. 4		CHANNEL No. 5	
	S	Fe	S	Fe	S	Fe	S	Fe	S	Fe
1.30 F	0.72	0.30	0.93	0.48	1.87	1.01	3.28	1.25	0.88	0.45
1.35 F	3.15	0.41	2.80	0.71	2.05	1.24	2.05	1.63	3.33	0.69
1.35 S	5.78	5.41	5.41	5.36	8.24	3.86	8.24	6.42	6.52	6.87
-24 M	0.86	0.57	1.60	1.23	1.91	1.47	4.15	2.14	1.56	1.56

One of the problems encountered was that, in a number of cases, the free iron had high values. This is apparent from the iron values tabulated in Appendix C. If total iron is high because of high free iron, the corresponding sulphur may still be low. These high values of free iron show the presence, in the coal, of iron other than pyritic iron and a relationship between total iron and sulphur becomes unreliable.

In view of the fluctuation in sulphur content, a statistical approach was taken with the data.

The percentage of organic sulphur in the coal sampled was found to be 0.65%, with approximate margin on bounds of error at ± 0.17 . For pyritic sulphur 1.54%, ± 0.48 . Total sulphur 2.19%, ± 0.51 . Expressed as a percentage of total sulphur, organic sulphur was 29.68% and pyritic sulphur 70.32%.

The correlation coefficient between total sulphur and pyritic sulphur over the height of the seam, for all samples, was 0.995, highly significant. The correlation between total sulphur and organic sulphur was 0.012, not statistically significant. This confirms the visual data in the histograms where the organic sulphur values over the height of the seam do not vary significantly and a change in total sulphur is the result of a change in pyritic sulphur.

The correlation coefficients were evaluated for a linear regression relationship between seam height and sulphur types. The values found for height and total sulphur was 0.661 and for height and pyritic sulphur 0.654, both statistically significant, and suggest a linear pattern of less total sulphur and pyritic sulphur as the height decreases. The values found for organic sulphur and height were not significant as already pointed out.

An analysis of variance could not establish significant variation in percentages of sulphur between samples taken and horizontal distances east-west and north-south.

The correlation coefficient for total sulphur in the plus 24 mesh fraction and sulphur in the 1.30 s.g. fraction was found to be 0.55 and for the 1.35 fraction 0.68, both statistically significant.

The possibility of a linear relationship between total sulphur and total iron was investigated. Correlation coefficients (r) and the regression coefficients (a) and (b) of the estimated relationship $S_t = a + b Fe_t$ for the different fractions are shown in Table 3.

TABLE 3

	-24M	1.30F	1.35F	1.35S
r	0.75	0.59	0.42	0.47
a	0.48	0.74	0.78	1.94
b	0.94	0.68	1.58	0.57
Bounds of Error	± 1.94	± 1.1	± 2.37	± 6.72

All correlations were significant, however the bounds of error in predicting total sulphur in terms of iron were large, and not very useful as a means of measuring sulphur during operations of the coal preparation plant.

The correlations coefficient for total sulphur and total iron for all fractions was 0.723. The regression equation was $S_t = 0.93 + 0.74 Fe_t$ and the bounds of error $\pm 2.76\%$.

TRACE ELEMENTS IN COAL

Trace elements in coal are of interest for at least three reasons:

1. In coal fired boiler plants, the deposition of trace elements and their compounds in plant equipment may reduce the overall efficiency of the plant.
2. Trace elements may interfere with catalytic activity in coal conversion.
3. Some trace elements are toxic to humans, animals, and plants, when they exist above certain concentrations. Other trace elements could prove to be carcinogenic, and effluent streams may have levels high enough to constitute a potential danger to the environment.

There is a definite lack of any systematic trace element analysis in Nova Scotia. Since coal utilization has emerged as an alternative source for Nova Scotia's energy needs, the effect of trace elements on industrial efficiency and the environment becomes an area requiring much needed study and research.

An early survey for trace elements in Nova Scotia coals was done by J.E. Hawley in 1955, using optical emission spectroscopy.⁴ The main purpose of this survey was to determine if a knowledge of trace element distribution could assist in the recognition and correlation of the individual seams making up the Cape Breton coalfield. Hawley concluded that in comparison with the ash of other coals, those of the Sydney coalfield are enriched in manganese, arsenic, and lead, have about the same amount of zinc and are lower in Be, B, V, Cr, Co, Ni, Ge, Mo, and Sn.

The most recent analytical report on trace elements was completed by M.R. Sandemar⁵, using neutron activation analysis. This report

suggests that trace element levels for coal samples from the Sydney coalfield are roughly comparable to other coal deposits in the United States, of similar rank. The possible exception being that there is a greater concentration of Mg and Hg as compared to certain Nova Scotia coals.

Table 4 compares the concentration ranges for sixteen Nova Scotia raw coals with those for various bituminous coals, as given in several publications.

Coal mining activities have the potential to effect the environment by land and water. Most Nova Scotia coal is extracted from underground mines, so that unsightly land areas, disturbed forest resources and the creation of large erosion surfaces, associated with strip mining, are not a major problem.

In view of the sulphur content of Nova Scotia coals with their acid producing potential, environmental concern is directed to the possible contamination of surface and ground water systems by controlled discharges of waste waters from mine sites. Due to their proximity to the ocean, all of the CBDC mines discharge untreated mine water either directly into salt water or into fresh water brooks which flow only short distances to reach the ocean. Typical water quality, including some trace elements, are summarized in Table 5. No deleterious effects have been documented in subtidal zones adjacent to discharge points and the effect on the marine receiving environment has been judged to be minimal.

CBDC's Victoria Junction wash plant generates the greatest volume of preparation plant waste in Nova Scotia. Course refuse is trucked to surface refuse piles and fines are pumped in a slurry to one of two tailing lagoons. The sulphur content of the refuse pile and lagoons is

TABLE 4

COMPARISON OF TRACE ELEMENT CONTENTS OF BITUMINOUS COALS
(ppm, unless otherwise indicated)

Element		Hawley HA55a	Ruch et al. RU77a	Block & Dams BL75b
Al %	0.24 - 4.00		0.31 - 3.1	0.67 - 3.8
As	5.9 - 281	3 - 270	0.34 - 120	0.5 - 25
Ba	< 15 - 240	2 - 257	5.0 - 1600	62 - 750
Br	5.8 - 75.1		0.5 - 52	3.2 - 24.4
Ca %	0.05 - 1.26		0.01 - 3.8	0.06 - 0.75
Ce	4.0 - 46		2.8 - 46	9.2 - 42
Cl	300 - 3520		100 - 8000	400 - 1400
Co	0.96 - 16	3.5 - 34	0.6 - 34	8.9 - 14
Cr	1.9 - 40	2 - 9	2.4 - 90	12 - 55
Cs	0.15 - 4.2		0.02 - 6.2	0.09 - 2.8
Dy	0.11 - 1.7		0.22 - 3.5	1.0 - 2.6
Eu	0.05 - 0.67		0.07 - 0.92	0.4 - 0.8
Fe %	0.66 - 6.47		0.30 - 4.1	0.08 - 1.1
Hf	0.17 - 1.7		0.13 - 2.2	0.3 - 1.8
Hg	0.012 - 0.10		0.02 - 1.6	0.5 - 0.6
I	0.34 - 2.8		0.20 - 14	1.3 - 4.1
In	< 0.003 - 0.036		0.01 - 0.63	0.03 - 0.06
K %	0.011 - 0.69		0.01 - 0.68	0.01 - 1.0
La	2.1 - 21.3		1.8 - 23	5.3 - 22.2
Mg %	< 0.02 - 0.23		0.01 - 0.39	0.15 - 1.2
Mn	19 - 266	22 - 254	1.4 - 220	14.1 - 300
Mo	< 0.4 - 15	2 - 12	0.10 - 30	1.2 - 2.4
Na	80 - 981		100 - 6000	300 - 1500
Rb	< 1.8 - 54		0.3 - 63	5.0 - 72
Sb	0.11 - 6.6		0.1 - 8.9	0.5 - 2.0
Sc	0.44 - 6.5		0.5 - 9.3	2.8 - 8.9
Se	< 0.7 - 6.7		0.4 - 8.1	1.0 - 2.0
Si %	0.35 - 6.0		0.58 - 6.3	0.73 - 7.9
Sm	0.27 - 2.7		0.22 - 4.3	1.5 - 3.7
Sr	< 13 - 1400	26 - 37	28 - 550	
Ta	< 0.04 - 0.18		0.04 - 1.1	
Tb	< 0.06 - 0.43		0.04 - 0.65	
Th	0.35 - 5.7		0.62 - 9.0	0.9 - 5.1
Ti	120 - 1400		200 - 1600	200 - 1700
U	0.51 - 0.97		0.30 - 4.6	
V	2.8 - 53	7 - 28	11 - 73	20 - 72
W	< 0.19 - 0.99		0.04 - 4.2	0.4 - 1.2
Yb	0.09 - 0.94		0.13 - 1.5	
Zn	< 4 - 470	13 - 64	0.30 - 5300	30 - 170

TABLE 5

TYPICAL QUALITY OF MINE WATER AND SURFACE RUNOFF
DISCHARGED TO THE OCEAN - SYDNEY AREA, N.S.

Parameter (total)	Total No. of Samples	Min. (mg/l)	Max (mg/l)	Average (mg/l)
pH	10	3.2	8.0	5.9
Acidity	5	22	150	61.6
Alkalinity	5	83	125	102
Susp. Solids	10	16	7,000	822
Diss. Solids	10	520	1,280	876
Sulphate	10	175	1,200	544
Al	10	0.5	20	6.7
As	3	0.02	0.14	0.06
Cd	10	0.001	0.069	0.026
Cu	10	0.001	0.17	0.045
Fe	10	0.58	30.0	7.98
Pb	10	0.006	0.34	0.17
Zn	10	0.01	0.72	0.22

Source: EPS-5-AR-79-5

concentrated in quantities sufficient to make it acid producing and could contain heavy metals which under acid conditions would be leached out. The concentration ranges of some elements in Sydney coal are illustrated in Table 6. The tailings lagoons are unlined and below the local water tables, accordingly, excess water and wash plant bleed, is ditched to a lime treatment facility prior to discharge into Northwest Brook. Samples of the tailings lagoon decant, coarse refuse pile run off and treated final effluent were collected by EPS Atlantic in 1977 and the analysis of these samples are shown in Table 7.

The increased use of coal as the fuel in electric generating stations will also increase the pollutants released to the environment. These pollutants are associated primarily with fuel storage on-site, by products of combustion and water used in operation of the steam cycle.

Coal contains a large number of chemical elements, including radio-nuclides, many of which concentrate in the ash upon combustion. A portion of these trace elements are dissolved in the ash transport water and are released to the environment with the direct discharge or seepage from the ash lagoon. Large waste water volumes can give rise to substantial total quantities being released to the environment over the lifespan of a generating station.

The elements found to leach most readily from coal ash include arsenic, barium, boron, cadmium, chromium, copper, selenium, and zinc.⁶ Radium in fly ash has also been found to be more soluble than that in soil.⁷

TABLE 6
MINOR ELEMENTS IN SYDNEY NOVA SCOTIA COAL AND ASH

	In Coal, ppm Average	Range	In Ash, ppm Average	Range
Arsenic	100	33 - 270	900	230 - 2300
Barium	34	2 - 257	300	18 - 2200
Beryllium	2	1 - 2	14	
Boron	17	6 - 25	148	52 - 220
Chromium	5	2 - 9	45	18 - 79
Cobalt	10	3 - 34	87	26 - 196
Germanium	5	1 - 8	44	9 - 70
Lead	66	25 - 120	572	
Manganese	140	9 - 254	1200	165 - 2200
Molybdenum	7	2 - 12	60	18 - 105
Nickel	15	6 - 74	131	52 - 645
Strontium	65	76 - 87	560	225 - 750
Tin	1		9	4 - 18
Vanadium	14	7 - 28	120	61 - 244
Zinc	25	13 - 64	218	115 - 550

Source: D.O.E. E.M.S. I.W.D. Atlantic, 1979.

TABLE 7

VICTORIA JUNCTION WASH PLANT TAILINGS LAGOON DECANT

Parameter (total)	Number of Samples	Min (mg/l)	Max (mg/l)	Average (mg/l)
pH	3	6.1	7.95	-
Suspended Solids	3	56	8700	2949
Dissolved Solids	3	2600	3600	3233
Sulphate	3	274	420	327
Acidity (CaCO ₃)	3	4	11	7
Copper	2	0.001	0.007	0.004
Cadmium	3	0.029	0.048	0.039
Iron	3	0.5	120.0	40.4
Lead	3	0.11	0.28	0.23
Zinc	3	0.01	0.40	0.16
Chloride	3	225	1880	1118
Aluminum	3	0.1	0.5	0.367
Cyanide	1	0.49	0.49	0.49

Source: EPS-5-AR-79-5

Chemical wastewaters at coal fueled power plants are usually diluted by release into the large volume cooling water discharge streams so that concern is not for the immediate or acute toxicity but the effect of large quantities of elements cumulating in the environment over the long-term.

One of the few publications available on trace elements is a study by E.L. Zodrow and M. Zentilli, which deals with the Uranium content of rock and coal in Cape Breton, Nova Scotia.⁸ Coal samples studied are on the average low in uranium content, about 0.5 ppm. Across a seam the uranium content appears to be at a minimum at the center and increases towards the hanging and footwalls. Zodrow's findings are illustrated in Table 8.

TABLE 8

Uranium content of coals from Sydney Coalfield arranged in stratigraphic sequence from oldest to youngest.

Location	Seam	Description or Remarks	U (ppm)		Standard deviation	Number of Samples
			mean	range		
1	Mc Aulay†	top of seam, outcrop	0.21			1
1	Mc Aulay	15cm below top of seam, outcrop	0.06			1
2	Shoemaker††	shaly coal	4.82	1.11-9.22	4.1	3
5	Tracy	from mine dump	2.15	0.08-4.22	2.9	2
8	Emery*	100 m of outcrop	0.43	0.03-3.18	0.7	23
12	Phalen	80 to 100cm below top of seam, outcrop	0.17	0.09-0.23	0.06	4
No. 16 Dominion	Phalen	former producer	0.04	0.03-0.05	0.01	4
1-B Mine (13)	Phalen	former producer	0.11	0.06-0.17	0.07	2
Lingan Mine	Harbour	present producer	0.82	0.05-1.65	0.80	3
Princess Mine	Harbour	former producer	0.24	0.14-0.35	0.15	2
Prince Mine	Stubbart	present producer	0.49	0.06-2.51	0.74	10
16	Upper Bonar	shaly coal	3.50*			1
Point Aconi	Point** Aconi	refer to Fig. 5.2	0.67*	1.1-0.2-0.7		3
AVERAGE FOR ABOVE COALS (excepting shaly coal)			0.46	0.03-4.2	0.80	55
AVERAGE FOR SHALY COALS			4.49	1.11-9.22	3.40	4
Wilkes Barre, Pennsylvania			0.45			1
Oberhausen, Germany			0.17			1
Siebengebirge, Germany			0.03			1
Measured stratigraphic thickness at sample site:						
† 35-40 cm			*150 to 180 cm		+Determined by DNA.	
†† 11 cm			**110 cm			
See Table 5.1 for information on the other seams.						

REVIEW OF WORK DONE ON SULPHUR REMOVAL
IN THE SYDNEY, CAPE BRETON, COALFIELD

REVIEW OF WORK DONE ON SULPHUR REMOVAL
IN THE SYDNEY COALFIELD

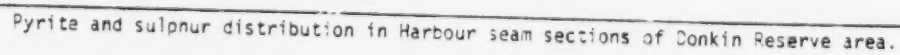
Cape Breton Development Corporation, CBDC, has long recognized the problem posed by the sulphur content in the Sydney coalfield. Over the years, considerable work has been done by and for Devco on cleaning of the coal, and predicting sulphur content for future mining.

Mention has already been made in this report of the work done by Dr. Agterberg on geomathematical predictions, and the work by J. Viseman on pilot plant studies leading to the construction of the present coal preparation plant. In 1978-79, it was decided to do exploration work on what was called "The Donkin Reserve Area."

This area is situated at the eastern extremity of the Sydney coalfield, and occupies an offshore area of 100 sq. km. Eleven boreholes were drilled within this area from a drillship and the boundaries have been drawn on the basis of 2.4 km. influence radii from the boreholes. The total insitu tonnage was calculated at 827,000,000 metric tons, of which 319,000,000 are in the Harbour seam.

A study was done on the intersections of the Harbour seam, and a petrographic profile of each intersection was constructed from conventional cores and sidewall cores.⁹ These profiles show the vertical reflectance, and also provide information on the predicted coke stability. Figure 8, from this study, shows the pyrite and sulphur distribution.

On the basis of this exploration work and including both the calculated reserves and known sulphur content, a decision was made to proceed with the Donkin Mine, presently under development.



GSC

Sandstone intrusions that exist in the overlying roof strata of the Sydney coalfield is the subject of an ongoing study by CBDC through its geological department. A continuous mapping program indicates that these sandstones are linear in nature and trend almost exclusively in a northeasterly direction. It is suggested that they are infilled paleo river channels which probably originated in the granitic provenance areas to the West and South and which eroded and migrated across the coal basin alluvial plain, sometime after peat deposition.

The existence of these channels, where they approach and intrude into the coal seam, cause instability in the roof, reduce the height of mineable coal, constitute a safety hazard when struck by steel mining picks, and increase the sulphur content of the coal seam.

Future occurrences of these channel sandstones can now be predicted with considerable confidence, in the short term. Extended and detailed study will be necessary to predict the direction and occurrences, in relation to the mining plan, for the long term. Reasonable predictions should be possible as data accumulates and controls for preferred direction are found.

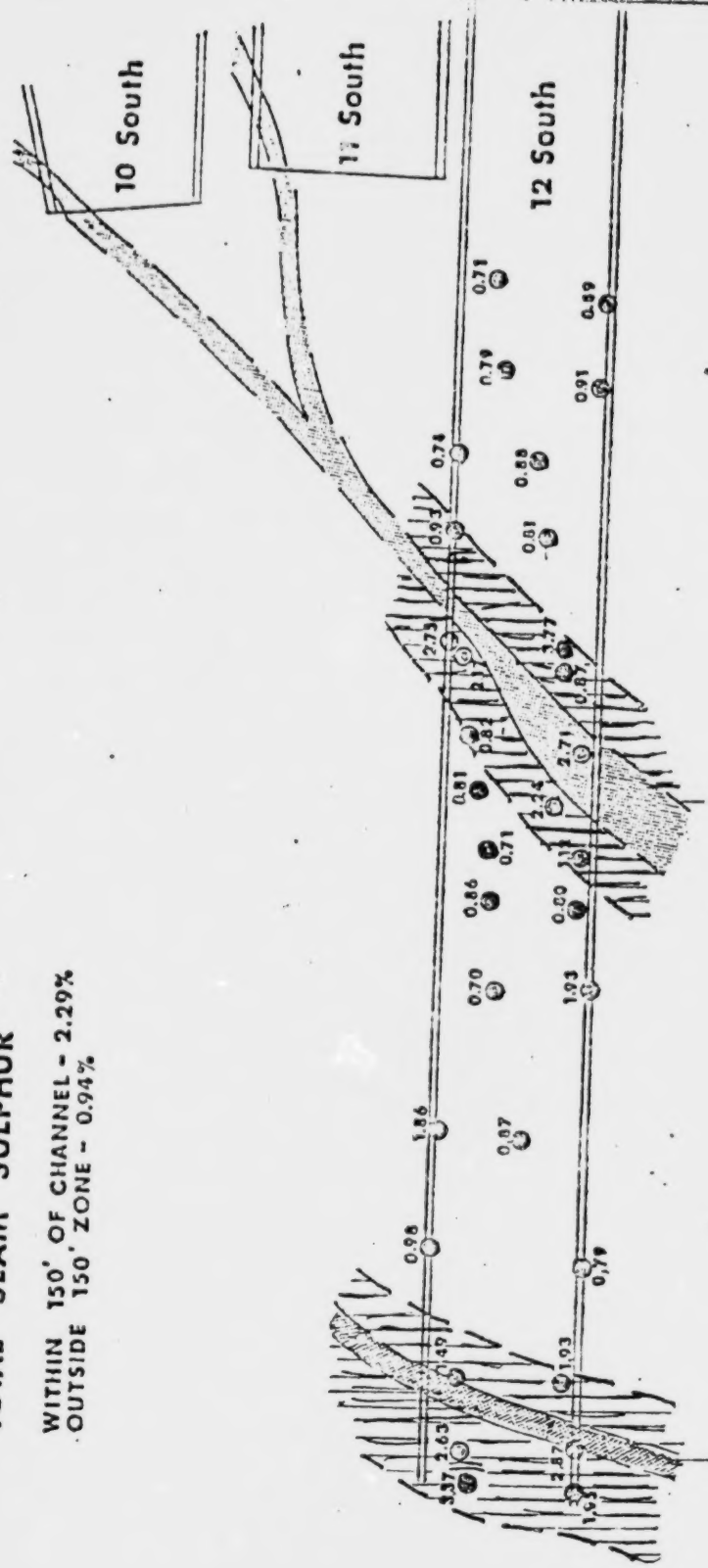
Figure 9 illustrates the effect on sulphur content in the coal seam where sandstone intrusions take place.¹⁰

CBDC has also received assistance from E.M.R. on flotation studies and N.S. Research Foundation on mathematical modelling for preparation plant operation.

Currently, CBDC is involved in sponsoring coal liquification (Synfuels Project), coal liquid mixtures (Carbogel project), Microbacteriological desulphurization, spherical agglomeration and column flotation.

TOTAL SEAM SULPHUR

WITHIN 150' OF CHANNEL - 2.29%
 OUTSIDE 150' ZONE - 0.94%



LEGEND

- SANDSTONE CHANNEL TREND (MAPPED)
- SANDSTONE CHANNEL TREND (PROJECTED)
- CHANNEL SAMPLE LOCATION & SULPHUR VALUE (TOTAL SEAM)
- 0 - 1.0% SULPHUR
- 1.0 - 1.5% SULPHUR
- 1.5 - 2.0% SULPHUR
- 2.0 - 2.5% SULPHUR
- 2.5 - 3.0% SULPHUR
- > 3.0% SULPHUR

FIGURE 9

CAPE BRETON DEVELOPMENT CORPORATION
 Geological Department
 PARTIAL PLAN OF #26 COLLIERY
 SHOWING
 SANDSTONE/SULPHUR CORRELATION

ATLANTIC GROUP FOR RESEARCH IN
INDUSTRIAL METALLURGY (AGRIM)

The Atlantic Group for Research in Industrial Metallurgy (AGRIM) was formed in October, 1974, to provide more effective support to local industry by pooling the scientific resources of various laboratories. It was also agreed that the most pressing metallurgical problem in Nova Scotia was the production of low sulphur steel from Cape Breton metallurgical coal.

The group was made up of the following:

1. Atlantic Regional Laboratories.
2. Nova Scotia Research Foundation Corporation.
3. Nova Scotia Technical College.
4. College of Cape Breton.
5. Sydney Steel Corporation.

Atlantic Regional Laboratories undertook to determine if it was economically feasible to remove additional sulphur by injecting desulphurized coke oven gas into a mass of coal as it pyrolyzed.

Typical blends of Cape Breton coking coals were pyrolyzed in a thirty pound coke oven while the flow rate and composition of the evolved gases were monitored by mass spectrometry. Injection of synthetic coke oven gas into a bed of coke caused further desulphurization at the higher temperatures of the process, but the extent of the desulphurization was not large enough to be important for present day industrial scale coke ovens. It was apparent that the pyrolytic emission of the H_2S was enhanced as the heating rate was increased up to 4° per minute. The results of further work indicated that about twice as much sulphur could be transferred from coal to coke oven gas if the heating rate could be increased about four times.

The Nova Scotia Research Foundation Corporation studied the transport and chemical reactions occurring as coal, previously extracted with ferric sulphate, is heated through the coking cycle. The results emphasized the need to evaluate the coking quality of coal desulphurized by ferric ion oxidation.

A further study was done on the potential of leaching to remove organic sulphur from Cape Breton metallurgical coal. Raw coal and ferric oxidized coal were treated and five classes of organic solvents were assessed. The tests indicated that 10 to 15 percent organic sulphur could be removed. Organic solvent leaching applied to ferric ion oxidized coal removed elemental sulphur but not organic sulphur. The organic sulphur content of coke produced from ferric ion oxidized Lingan coal is 35 to 54 percent less than organic sulphur content of coke produced from untreated coal. Insoluble iron sulphate was successfully removed in excess of 90 percent by aspirating a liquid vortex from the coal, the finely dispersed sulphate being in suspension within the vortex.

The problem of reconstituting coal fines subsequent to chemical extraction of sulphur was investigated with considerable success by incorporating fibres of cellulose with the wet coal. The results justify more work on this approach to the reconstitution of fine coal. N.S.R.F.C. is presently working on a coal liquefaction project, have recently completed a project on the pyrolysis of coal-line mixtures, and coal desulphurization by the elimination of magnesium ion effect.

Nova Scotia Technical College studied the effects of rare earth metal additions on the microstructure and mechanical properties of

rail steels, particularly through the shape control of sulphide inclusions. A significant improvement in impact strength, especially in the transverse direction, was noted in the rare earth metal treated rails. The effect of rare earth additions on the mechanical properties of railroad axle steel was also investigated and considerable improvement in the fatigue strength was found in the treated steels.

Presently T.U.N.S. is investigating the temperature range over which pyrite transforms to pyrrhotite in the coking of Prince coal. Expected to complete March, 1983.

The College of Cape Breton did thermoanalytical work on Cape Breton coal which provided a general view of the sequence of events involved in the thermo-decomposition of coal from No. 26 Colliery. Indications were obtained that changes occur in the coal at temperatures as low as 200°C. The formation of asphaltenes was found to occur predominantly at this low temperature region and evidence was found in support of the proposal that asphaltenes are an intermediate in the conversion of coal to coke. Thermal analysis was also used to follow possible changes in the thermal decomposition of coals that had been leached in the Meyers process, but no alterations were detected.

Sydney Steel Corporation concentrated its efforts toward the development of a safe and efficient method of desulphurizing fifty ton ladles of molten pig iron from the Blast Furnace. An experimental injection station was built adjacent to the Open Hearth Shop and was used to lance inject powered desulphurizing agents such as magnesium and calcium based materials into the ladles. A number of parameters were established, such as material flow rate, lance design, and fluidizing tank and line pressures. The actual injection of the agents proved to be an efficient method except for the uncontrollable violent splashing of the hot pig iron metal over

the top of the ladle. Bulk additions of calcium based material to the ladle was also tried but proved to be inefficient for removing sulphur.

Mag-coke, a porous coke impregnated with magnesium, when plunged below the slag layer of the molten pig iron, proved to be the most efficient method of desulphurizing within the confines of a fifty ton ladle.

PREPARATION PROCEDURES AND CONCEPTS
APPLICABLE TO NOVA SCOTIA COALS

PREPARATION PROCEDURES AND CONCEPTS
APPLICABLE TO NOVA SCOTIA COALS

Currently, the available methods for reducing sulphur emissions from coal fired sources fall into the following:

1. The use of low sulphur coal, either naturally occurring or physically cleaned.
2. Stack gas scrubbing.
3. Chemical treatment to extract sulphur from coal.
4. Conversion of coal to clean fuel by gasification or liquefaction.
5. Coal, oil, water mixtures.
6. Fluidized bed concepts.

Physical cleaning of coal has the most developed technology and the lowest cost of the available methods for reducing emissions. Sulphur in coal exists principally in two forms, organic and inorganic (pyritic). Organic sulphur can not be physically removed but pyritic sulphur can be removed by crushing and cleaning. The degree of removal is dependent on pyrite size, and distribution, coal size, and other physical characteristics.

The present CBDC coal preparation plant was designed primarily to produce a metallurgical coal, and because the thermal coal specifications were less stringent with respect to sulphur, the rejects from the metallurgical coal circuit could be treated to produce a thermal grade product and a final discard. The input sulphur which has wide fluctuations largely determines the metallurgical - thermal coal ratio.

It is recognized that the pyrite in Nova Scotia coal is finely disseminated and would require fine grinding in order to liberate increased amounts and reduce sulphur. The present plant operation, with crushing to

one and one half inches, cannot effectively reduce pyritic sulphur and it becomes an exercise in redistributing sulphur, by specific gravity separation, providing a metallurgical coal at approximately 1.2% sulphur and thermal coal that still contains 2.5 - 3.5% sulphur. Although the refuse from the present plant is approximately 15%, only about 3% is coal loss mostly from the fine tailings. It would be possible to adjust the gravity cut off point for the thermal product but this would correspondingly increase the coal loss. In view of the necessity for an economic balance in the preparation plant and the retained organic sulphur, it is extremely unlikely that the guidelines could be met for sulphur emissions, and a further method must be found at the coal fired source.

High Gradient Magnetic Separation as a dry process has been successfully demonstrated. This technique beneficiates the coal by subjecting it in a finely pulverized form to a high-intensity magnetic field. The dry separation reduces the problems and the costs associated with dewatering fine coals. CANMET'S Mineral Processing Laboratory has done work with this technique on Lingan and Prince Mine coals.¹¹ Reductions of 91.3% ash and 69.7% total sulphur for Lingan coal and 78% ash and 67.1% sulphur for Prince coal were achieved. More research in this area is indicated and an economic assessment of heavy media separation compared to magnetic separation should prove interesting.

FLUE GAS DESULPHURIZATION

Most of the sulphur in the coal is oxidized to SO_2 and emitted with the combustion gas. The scope of the problem can be appreciated if we consider that a 3.5% sulphur coal results in an emission exceeding 600 tons of SO_2 per day from a 1000-MW boiler.

Use of the wet scrubbing operation with lime or limestone is the most popular method of removing SO_2 from waste gas. A simplified flow sheet is shown in figure 10. The makeup lime or limestone is added to a tank in the main scrubber circulation loop. The scrubbed gas is passed through a mist eliminator to knock out mist droplets entrained in the gas stream and reheated to avoid condensation in the stack. Part of the product slurry is bled off from the hold tank and sent to a pond where the solids settle out. The choice between lime and limestone must be evaluated in any installation. In general, limestone is cheaper but not as reactive or as easy to use effectively in the scrubber. The quantity of waste solids generated when using high-sulphur coal is quite large and the usual practice is to use a disposal pond as indicated in figure 10.

The achievable sulphur reduction by physical cleaning will define the level of sulphur prior to combustion and will therefore define the required degree of flue gas desulphurization to meet environmental standards. The degree of desulphurization required can be achieved by employing either a low removal efficient system or a highly efficient system treating only a portion of total stack gas.

A case was made for the combined approach of physical coal cleaning (PCC) followed by flue gas desulphurization (FGD) by the United States Environmental Protection Agency.¹² The U.S. study concluded that in many cases the net cost of PCC followed by FGD is substantially less than that of FGD alone. The key element in showing the advantage in favor of the combined approach is the difference in FGD costs. This difference results from only 21% of flue gas being processed by the FGD system as opposed to 85% when clean coal is not used.

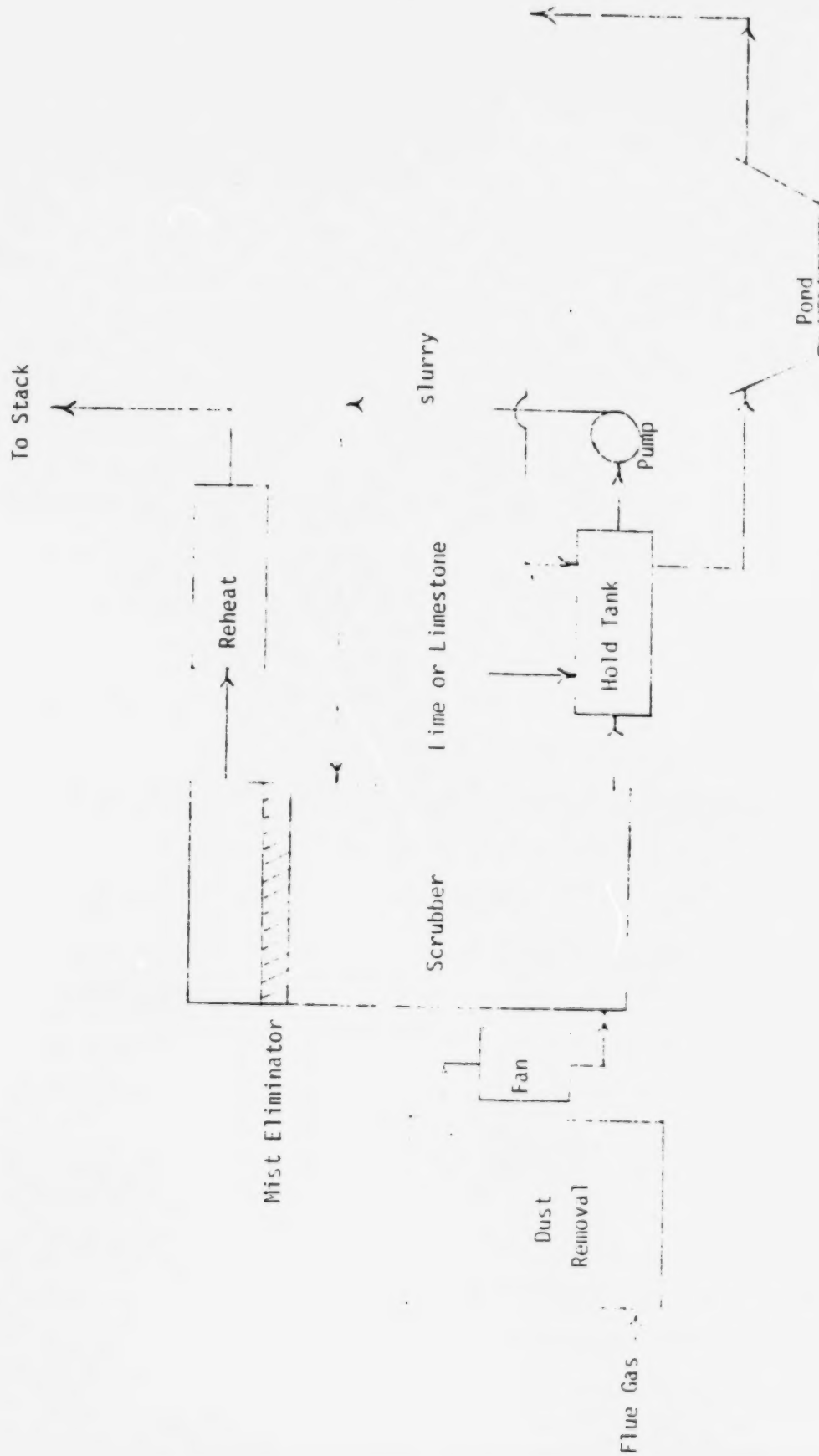


Figure 10
Simplified Flowsheet of
Lime-Limestone Scrubbing

Control of stack emissions by FGD is expensive, it has been estimated that an installation to control stack emissions including particulates, accounts for 20-25% of the capital cost of a new power station. Retrofit scrubbers usually cost 10-20% more than scrubbers in new installations.

CHEMICAL COAL CLEANING (CCC)

The chemical removal of sulphur from coal requires that coal particles be treated with reagents under physical conditions of temperatures and pressure that will allow the removal of sulphur without thermal degradation of the coal substance itself. A variety of chemical coal cleaning processes are now being developed which will remove a majority of the pyritic sulphur and some are capable of removing a percentage of organic sulphur. A list of the major processes would include Magnex, Syracuse, TRW and Legemont, which will remove pyritic sulphur only. Processes which will remove most of the pyritic sulphur and varying amounts of organic sulphur would include, ERDA, GE, Battelle, JPL, IGT, KVB and ARCO.

Chemical coal cleaning processes are still under development and none have been tested in large units. The TRW (Meyers) process is probably the most advanced. It is a chemical leaching process using ferric sulfate and sulfuric acid solution to remove the pyritic sulphur from coal. This process has removed 80 to 96% of pyritic sulphur from 14 mesh top size coal. The Gravicheme process is a variant of the Meyers process. The iron sulfate/sulfuric acid leach solution can be used as a heavy medium to gravity-separate fine coal of specific gravities of 1.2 - 1.35. The portion of the coal that floats in the leach solution is almost pyritic

free and can bypass the meyers sulphur extraction process, offering overall cost improvements.

The ERDA process is probably the most advanced in removing organic as well as pyritic sulfur. It is an air/steam leaching process, in which the coal slurry is heated under pressure, resulting in the removal of all the pyritic sulphur and 40% of the organic sulphur, in the form of sulfuric acid.

In general, chemical desulphurization processes are higher in capital investment and annual revenue requirements than physical coal cleaning. Research is ongoing, and a breakthrough could make chemical desulphurization technics attractive, especially when integrated with physical coal cleaning.

Gasification of Coal may be classified into three categories depending on the heating value of the product gases.

1. Low - BTU gas will normally have a heating value in the range 100-200 BTU's per standard cubic foot. The combustible components consist of CO and H₂ diluted by CO₂ and N₂. Its use is relatively limited.
2. Medium - BTU gas has a calorific value between 270 and 600 BTU/SCF. At the lower range the gas consists primarily of CO and H₂ with small amounts of CO₂. At the higher end of the range the heating value is increased by the inclusion of methane (CH₄). This gas is very useful in manufacturing industries because it burns rapidly and produces a flame temperature as high or higher than natural gas.
3. High BTU gas is synonymous with natural gas and has a heating range of 970 to 100 BTU/scf., its composition is mainly methane with small amounts of CO, CO₂ and N₂.

The benefits of gasification of coal are that the products will be clean burning fuels and environmentally compatible, as well as easily stored and transported. The technology status of coal gasification processes in the United States is shown in Table 9.

Underground gasification is a method of converting coal, without mining, into combustible gases, by initiating combustion in a coal seam and injecting a current of air, oxygen, or oxygen and steam. All processes require inlet and exhaust holes from the surface. The Sydney coal field is located under the ocean and would require offshore drilling and costly pipeline installations. In situ gasification does not appear to be a viable option in the near future.

Liquification of coal is the conversion of large organic molecules of the coal into liquids and gases by the addition of hydrogen or the elimination of carbon. When the hydrogen ratio in coal is increased by whatever means it will liquify. There is a wide array of process technology but it can be broken down into four categories within two larger divisions of direct and indirect liquefaction.

Direct Liquefaction:

1. Pyrolysis - these processes use thermal decomposition of the organic components of the coal in the absence of oxygen to produce a solid char, along with liquids and gases.
2. Solvent Extraction - a solvent is used as a hydrogen donor to promote liquefaction under high temperature and pressure.
3. Direct Hydrogeneation - these are processes in which coal is catalytically hydrogenated under high temperature and pressure.

TABLE 9
TECHNOLOGY STATUS OF COAL GASIFICATION PROCESSES

Process (developer)	Product gas Btu content	Reactor bed type	Number of stages	Pressure/temperature (atm/°C)	Current capacity (t/c)	Possible commercialization
Commercial						
Koppers-Totzek (Koppers GmbH)	Medium	Entrained	1	1-550-2150	350	Available now
Lurgi (Lurgi Minerals AG, Germany)	Medium	Moving	1	20-30/60-750	1000	Available now
Wellman-Ludlow (Wellman)	Medium	Moving	1	1/600	101	Available now
Winkler (Bayer Powergas)	Medium or low	Fluidized	1	1-3/350-1000	1500	Available now
Development						
Lurgi - using agglomerating coal	Low	Moving (stirred)	1	20/500	2000	By 1965
Pilot Plant						
U-GAS (Bituminous Coal Research)	Medium	Entrained	2	70-100/900-1500	120	By 1965
Cojet (MCC)	Medium	Fluidized	1	2-4/900	50 (char)	Near 1965
AFRC (AFRC/Consolidated Coal)	Medium	Fluidized	2	10-20/900	40	Near 1965
U-GAS (Institute of Gas Technology)	Medium	Fluidized	2	70-100/700-1000	80	By 1965
Stirling (Pittsburgh Energy Res. Ctr.)	Medium	Fluidized	1	40-70/900	75	By 1965
Union Carbide/Battelle	Medium	Fluidized	1	7/1000	25 (H.C.)	Near 1965
Stirred fixed bed reactor (Pittsburgh Energy Res. Ctr.)	Low	Moving	1	20/550	20	Near 1965
Consolidated Edison						
Consolidated Edison	Low	Entrained	1	1-10/1150*	120	After 1965
Westinghouse	Low	Fluidized	2	10-15/900-1000 and 1150	15	Near 1965
Pittsburgh & Midway Coal Mining	Low	Entrained	2	4-35/1150*	30	Near 1965
U-GAS (Institute of Gas Technology)	Low	Fluidized	1	20/1050	20	After 1965
Laramie Energy Research Center	Low	Underground (in situ)	1	10/2	15-45	Near 1965

(SOURCE: 102, p. 9)

Indirect Liquefaction:

These processes use the gasification of coal with steam and oxygen to produce synthesis gas which is catalytically converted to liquid products.

The status of selected coal liquefaction processes in the United States is shown in table 10.

It is generally agreed that the optimum capacity to make a plant viable is a production of 100,000 bbl./day of product and such a plant would consume coal at the rate of 30-40,000 tons per day. This consumption of coal is obviously beyond the production rate for Nova Scotia coal mines in the near future. The continuing development of various processes and technology may make it possible that a scaled down version might become a viable option. Scotia synfuels a consortium of Federal-Provincial-Crown corporations and private capital have indicated their intention to proceed with a plant at some time in the future.

TABLE 10

COAL LIQUEFACTION PROCESSES

PROCESSES	DEVELOPMENT STATUS	DEVELOPMENT PLANS	POSSIBLE COMPLETION DATE
Solvent Refined Coal	Pilot plant, 50 t/d	Demonstration plant operational FY 83	After 1985
H-Coal	PDU, 2.5 t/d	600 t/d Pilot plant operational FY 79	Mid. 1985
Exxon Donor Solvent	PDU, 1 t/d	250 t/d Pilot plant operational FY 83	After 1985
Synthoil	Bench Unit, 0.5 t/d	10 t/d PDU operational FY 79	After 1985
CC-2	Pilot plant, 16 t/d	None projected	After 1985
Co-Stream	CPU		
Clean Coal	PDU, 0.5 t/d	None in this country	
Fluor-Tripath	Fundamental research		
Ox-C (Garrett)	PDU, 3 t/d	250 t/d Pilot plant	
Calder	Re-evaluation		
Merthol Synthesis			
TOPCOAL	data unavailable		
Beagles	bench unit, 0.5 t/d	PDU	Near 1985, unless cancelled

(SOURCE: IGT, p. 10)

COAL-OIL-WATER MIXTURES

Coal-oil mixtures may give an economical means of extending the life of existing oil-fired facilities, especially where full conversion to coal would impose a substantial derating and high conversion cost. This is due to the fact that coal-oil mixtures have handling, storage and combustion characteristics similar to No. 6 oil.

Coal-oil-water mixtures can be blended and adjusted to influence the amount of SO_2 emissions to some degree, but the process would gain in importance should the CBDC consider fine grinding to liberate pyrite, in their preparation plants. Indications are that the optimum coal particle size for coal-oil mixtures is 70-90% through 200 mesh.

The economics for a particular set of conditions, boiler capacity, fuel prices, conversion cost, etc., need to be assessed, but the consensus appears to be that coal-oil or coal-oil-water mixtures are a near term alternative to fuel oil firing in boilers.

The Center for Energy Studies at the Technical University of Nova Scotia has done fundamental studies on coal separation, flotation, stability factors and development work on flame and combustion evaluation along with beneficiation to produce coal-oil mixtures using Nova Scotia coals.

Scotia Liqui-Coal Ltd. also did development work to produce coal-oil mixtures using Nova Scotia coals and this project is now at the pilot plant stage.

A process originating in Sweden has aroused considerable interest. This is a coal-water mixture known as Carbogel. Present plans call for the production of this material at CBDC Coal Preparation Plant in Cape

Breton and testing at the New Brunswick Electric Power Commission at Chatham, New Brunswick.

Fluidized Bed Combustion can be considered a further method for reducing sulphur emissions from coal fired sources. This process is not new, it has been used by the chemical processing industry for half a century. A more recent interest in its use in the field of steam generation has been initiated because the coal can be burned without producing environmental restricted SO_2 emissions.

A fluidized bed system consists basically of a water cooled enclosure, air distribution grid, boiler tubes immersed in the fluidized bed during operation, a method to introduce the coal and bed material, and a means of removing ash and spent bed material. The bed is formed of sand, crushed refractory, limestone or coal ash and is heated by start-up burners. When air is passed upwards through the bed, the particles move further apart as the velocity increases until the total weight of the bed is supported and takes on the properties of a boiling liquid. Coal introduced to the bed burns rapidly and the heat is transferred to the bed material, which in turn transfers the heat to the immersed boiler tubes. If the material in the bed is limestone it is converted to calcium oxide during the burning process. Sulphur in the coal burns to sulphur dioxide and combines with the calcined limestone to form gypsum. The sulphur is thus contained in the solid form which is easier to handle and dispose. The temperatures of combustion are between 1500 to 1600°F (816 to 870°C) which also greatly reduces the formation of oxides and nitrogen.

The advantages of this process are:

- (a) Versatility of fuel choice, high sulphur, high ash coal may be used.
- (b) Low emission levels of SO_2 and NO_x , conforming to environmental standards.
- (c) High heat transfer resulting in more compact boiler design and lower capital costs.
- (d) Low combustion temperature resulting in greater operational reliability and lower maintenance costs.
- (e) Coal preparation costs reduced. Fluidized bed process will accept larger size coal feed.

Fluidized bed combustion in boiler sizes up to 100,000 lb/hr. has already been successfully proven. The application of larger units for utility plant operations are still in the development stage. It is expected that this technology will be proven before the end of the 1980's.

The advantages of the CBDC operated Sydney coalfield are obvious, since the Preparation Plant could then be operated to produce a metallurgical grade coal and the rejects used in fluidized bed combustion which could satisfy the environmental standards for emissions.

Indications are that a fluidized bed installation may be in the future for the Nova Scotia Power Corporation's generating plant at Port Hawkesbury.

A P P E N D I X A

STANDARD TEST METHOD FOR FORMS
OF SULPHUR IN COAL



AMERICAN NATIONAL
STANDARD

ANSI/ASTM D 2492 - 77

Standard Test Method for FORMS OF SULFUR IN COAL¹

This Standard is issued under the fixed designation D 2492; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method² covers the determination of the three commonly recognized forms of sulfur in coal: sulfate sulfur, pyritic sulfur, and organic sulfur.

2. Applicable Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water³

D 3177 Test for Total Sulfur in the Analysis Sample of Coal and Coke⁴

3. Summary of Method

3.1 Sulfate sulfur is determined by extracting a weighed sample of coal with dilute hydrochloric acid followed by precipitation with barium chloride (BaCl_2) and weighing as barium sulfate (BaSO_4). Sulfate sulfur is soluble in dilute hydrochloric acid; pyritic and organic forms of sulfur are insoluble.

3.2 Pyritic sulfur is determined by extracting a weighed sample of coal with dilute nitric acid followed by titrimetric or atomic absorption determination of iron in the extract as a measure of pyritic sulfur. Extraction of pyritic sulfur from coal with dilute nitric acid involves oxidation of ferrous iron to ferric and sulfide sulfur to sulfate, both of which are soluble in nitric acid. Because the extraction dissolves sulfate and pyritic sulfur plus a small amount of organic sulfur, the dissolved sulfur is not a reliable measure of pyritic sulfur. Consequently, it is obtained by determining the amount of iron combined in the pyritic form, which is equal to the difference between nitric acid and hydrochloric acid-soluble iron.

3.2.1 The sample of coal used for the pyritic sulfur determination may be a separately

weighed sample or the residue from the hydrochloric acid extraction for sulfate sulfur. In the latter case, two acid extractions are carried out on the same sample, the nitric acid treatment being applied to the coal residue from the hydrochloric acid extraction for determination of sulfate sulfur. Determination of iron in the hydrochloric acid extract is unnecessary, because iron in the nitric acid extract represents pyritic iron. However, there are certain limitations to the use of the sulfate sulfur residue for determination of pyritic sulfur in coal: if pyritic iron is high, the large sample required for determination of small amounts of sulfate sulfur will contain large quantities of iron and may require dilution; the determination of pyritic iron cannot be carried out until both extractions of sulfur have been completed.

3.3 Organic sulfur is determined by subtracting the sum of sulfate sulfur and pyritic sulfur from the total sulfur as determined in accordance with Method D 3177.

4. Apparatus

4.1 Balance, sensitive to 0.1 mg.

¹ This method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved June 24, 1977. Published August 1977. Originally published as D 2492 - 66 T. Last previous edition D 2492 - 68 (1974).

² For information concerning experimental work on which this method is based, see the following papers:

Powell, A. R., "The Analysis of Sulfur Forms in Coal," Technical Paper No. 254, U. S. Bureau of Mines, 1921.

Krumm, P. O., "The Third Interlaboratory Study of Methods for the Determination of Total Sulfur, Forms of Sulfur, and Chlorine in Coal," Report of Investigation, OESRA, Ohio State University Engineering Experiment Station, 1960.

³ Annual Book of ASTM Standards, Parts 20, 21, 22, 26, 29, 31, 37, 40, and 43.

⁴ Annual Book of ASTM Standards, Part 26.

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4.2 *Cold-Finger Condenser*, to fit a 250-ml Erlenmeyer flask.

4.3 *Crucibles*—Porcelain, silica, or platinum crucibles of 10 to 15-ml capacity shall be used for igniting BaSO_4 .

4.4 *Hot Plate*—Electric or gas-heated hot plate, with full temperature control permitting adjustment of surface temperature.

4.5 *Muffle Furnace*—Electrically heated muffle furnace capable of maintaining the temperature at approximately 925°C .

4.6 *Atomic Absorption Spectrophotometer*.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.¹ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

5.3 *Ammonium Chloride* (NH_4Cl), solid.

5.4 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

5.5 *Barium Chloride Solution* (100 g/litre)—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 litre.

5.6 *Diphenylamine Sulfonate Indicator Solution* (0.20 g/100 ml)—Dissolve 0.20 g of sodium or barium diphenylamine sulfonate in 100 ml of water. Store in a dark colored bottle. This reagent is required only when $\text{K}_2\text{Cr}_2\text{O}_7$ solution (5.16) used as a titrant for iron (6.2.3.1).

5.7 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.8 *Hydrochloric Acid* (2 + 3)—Mix 2 volumes of concentrated HCl (sp gr 1.19) with 3 volumes of water.

5.9 *Hydrochloric Acid* (1 + 23)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 23 volumes of water.

5.10 *Hydrogen Peroxide* (30 %)—Concen-

trated hydrogen peroxide (H_2O_2).

5.11 *Iron Standard for Atomic Absorption*—Clean a length of pure iron wire and weigh exactly 1 g to the nearest 0.1 mg. Place in a 250-ml beaker, add 100 ml of HCl (1 + 1), and cover the beaker with a watch glass. When the iron is all in solution, transfer the solution and beaker washings to a 1000-ml volumetric flask and dilute to volume.

5.12 *Lanthanum Solution*—Dissolve 175 g of LaCl_3 or 265 g of $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$ in water and dilute to 1 litre. Alternately slurry 115 g of La_2O_3 in 200 ml of water, slowly add 200 ml concentrated hydrochloric acid to dissolve the oxide. Dilute to 1 litre.

5.13 *Mercuric Chloride Solution (Saturated)*—Dissolve 8 g of mercuric chloride (HgCl_2) in 100 ml of hot water, cool to room temperature, and filter.

5.14 *Methyl Orange Indicator Solution* (0.02 g/100 ml)—Dissolve 0.02 g of methyl orange in 100 ml of hot water and filter.

5.15 *Nitric Acid* (1 + 7)—Mix 1 volume of concentrated nitric acid (HNO_3 , sp gr 1.42) with 7 volumes of water.

5.16 *Potassium Dichromate Solution* (0.0500 N)—Twice recrystallize potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) from water. Dry the crystals at 110°C , pulverize, and dry at 180°C to constant weight. Dissolve 2.4518 g in water and dilute to 1 litre with water. This is a primary standard (Note 1). This reagent is required only when $\text{K}_2\text{Cr}_2\text{O}_7$ solution is used as a titrant for iron (6.2.3.1).

Note 1—National Bureau of Standards oxidimetric standard sample No. 136 of $\text{K}_2\text{Cr}_2\text{O}_7$ is satisfactory for this purpose. When this standard is used, recrystallization is unnecessary.

5.17 *Potassium Permanganate Solution* (0.05 N)—Accurately standardize a 0.05 N solution of potassium permanganate (KMnO_4) against a primary standard (Note 2), using four decimal places.

Note 2—National Bureau of Standards standard sample No. 55d of ingot iron is satisfactory for this purpose.

¹ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."



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5.18 Stannous Chloride Solution (5 g/100 ml)—Dissolve 5 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 10 ml of concentrated HCl (sp gr 1.19) and dilute to 100 ml with water. Prepare this solution fresh before use.

5.19 Sulfuric-Phosphoric Acid Mixture ($\text{H}_2\text{SO}_4\text{-H}_3\text{PO}_4$)—Slowly add 150 ml of concentrated H_2SO_4 (sp gr 1.84) and 150 ml of concentrated H_3PO_4 (85 %) to 700 ml of cold water, while stirring constantly. This reagent is required only when $\text{K}_2\text{Cr}_2\text{O}_7$ solution (5.16) is used as titrant for iron (6.2.3.1).

5.20 Sulfuric-Phosphoric Acid-Manganous Sulfate Mixture—Dissolve 75 g of manganous sulfate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) in 500 ml of cold water, slowly add 150 ml of concentrated H_2SO_4 (sp gr 1.84) and 150 ml of concentrated H_3PO_4 (85 %) and dilute to 1 litre with water. This reagent is required only when KMnO_4 solution (5.17) is used as titrant for iron (6.2.3.1).

6. Procedure

6.1 Sulfate Sulfur:

6.1.1 Extraction of Sulfate Sulfur—Weigh to the nearest 0.1 mg about 5 g of thoroughly mixed air-dried coal which has been ground to pass a No. 60 (250- μm) sieve, transfer to a 250-ml Erlenmeyer flask, and add 50 ml of HCl (2 + 3). Fit the cold-finger condenser into the neck of the flask, place on the hot plate, and boil for 30 min. Rinse the cold-finger condenser into the flask with HCl (1 + 23) and filter through a medium-texture, double acid-washed filter paper into a 400-ml beaker. Transfer the residue to the filter, wash six times with HCl (1 + 23) using a total of about 35 ml, and determine sulfate sulfur in the filtrate as described in 6.1.2 and 6.1.3. The residual coal may be retained for the determination of pyritic sulfur. Discard the residual coal if a separate sample is used for the pyritic sulfur determination.

6.1.2 Removal of Iron—To the filtrate from 6.1.1 add 10 ml of saturated bromine water, cover with a watch glass, and boil for 5 min to ensure that all iron is in the ferric state and to expel excess bromine. Precipitate iron by slowly adding NH_4OH (sp gr 0.90) until a slight excess is present, then add 5 ml more, constantly stirring to coagulate the yellowish brown ferric hydroxide precipitate. Place the

covered beaker on the hot plate and boil for 1 min. Filter off the iron precipitate using an ashless, medium-texture filter paper, collecting the filtrate in a 600-ml beaker. Wash the precipitate several times with hot water to which has been added a trace of NH_4Cl and NH_4OH . Retain the filtrate, with washings, for determination of sulfur, and the precipitate, with the original beaker, for the determination of nonpyritic iron when a separate sample is used for the determination of pyritic sulfur. If a separate sample is not used, the precipitate may be discarded.

6.1.3 Final Determination of Sulfate Sulfur—Neutralize the filtrate from 6.1.2 with concentrated HCl (sp gr 1.19) and add an excess of about 1 ml, employing methyl orange as indicator. Heat the solution to boiling and add dropwise, with stirring, 10 ml of the BaCl_2 solution. Continue boiling for 15 min and allow to stand for 2 h just below boiling. Filter through an ashless paper and wash with hot water until silver nitrate (AgNO_3) solution shows no precipitate with 1 drop of the filtrate. Place the wet filter containing the precipitate of barium sulfate (BaSO_4) in a weighed platinum, porcelain, silica, or refractory crucible, allowing a free access of air by folding the paper over the precipitate loosely to prevent spattering. Smoke the paper off gradually and at no time allow it to burn with flame. After the paper is practically consumed, raise the temperature to approximately 925°C and heat to constant weight. The value so obtained, corrected for the blank (see 6.1.4) represents the sulfate sulfur of the coal.

6.1.4 Sulfate Blank—Make a blank determination following the same procedure and using the same amounts of all reagents as described in 6.1.1, 6.1.2, and 6.1.3.

6.1.5 Determination of Nonpyritic Iron—This determination must be made if a separate sample is used for determination of pyritic sulfur. If the residue from the HCl extraction is used for determining pyritic sulfur it is not necessary. Dissolve the iron hydroxide precipitate (6.1.2) and determine iron in the solution as described for pyritic iron in 6.2.3.1 or 6.2.3.2.

6.2 Pyritic Sulfur:

6.2.1 Extraction of Pyritic Sulfur—The ex-

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traction of pyritic sulfur may be carried out on the residue from the HCl extraction of sulfate sulfur or on an accurately weighed sample of approximately 1 g of the original coal.

6.2.1.1 Transfer the sample to a 250-ml Erlenmeyer flask and add 50 ml of HNO_3 (1 + 7). Fit a cold-finger condenser in the neck of the flask, and either boil for 30 min or let stand overnight at room temperature. Rinse the cold-finger condenser with HNO_3 (1 + 7), filter the contents of the flask through a medium-texture, double acid-washed filter paper, and wash six times with the HNO_3 (1 + 7), collecting the filtrate and washings in a 600-ml beaker. Discard the residual coal on the filter paper and retain the filtrate for determination of iron which is a measure of pyritic sulfur.

6.2.1.2 Add to the filtrate 2 ml of 30 % H_2O_2 and boil for 5 min to destroy any coloration arising from decomposition of the coal.

NOTE 3—Somewhat more organic matter may be brought into solution when boiling subbituminous coals, lignites, and brown coals with nitric acid. This may be destroyed by evaporating the filtrate to dryness, adding concentrated sulfuric acid, and heating to fumes of sulfur trioxide.

6.2.2 *Separation of Iron for Titration*—Heat the filtrate (6.2.1.2) to the boiling point and precipitate iron by slowly adding NH_4OH until a slight excess is present, stirring constantly to coagulate the ferric hydroxide. Place the covered beaker on the hot plate and boil for 1 min. Filter through an ashless, medium-texture filter paper into a beaker and wash the precipitate six times with hot water to which has been added a trace of NH_4Cl and NH_4OH . Retain the precipitate for the determination of iron in accordance with 6.2.3.

6.2.3 *Determination of Pyritic Iron by Titration*—Dissolve the iron hydroxide precipitate and determine iron in the solution as described in 6.2.3.1. If the residue from the sulfate sulfur extraction is used for this determination, the iron represents pyritic iron and thus pyritic sulfur. If a separate sample is used, the iron represents both nonpyritic and pyritic iron and must be corrected for nonpyritic iron to be representative of pyritic sulfur.

6.2.3.1 *Determination of Iron by Titration*—Dissolve the iron hydroxide precipitate from the filter paper with 15 to 25 ml of hot

HCl (2 + 3) and wash the filter thoroughly with hot water. Heat the solution to boiling. Add SnCl_2 solution dropwise, while stirring, until the color of the ferric iron is discharged, and then add 1 or 2 drops excess but no more. Wash down the inside of the beaker and quickly cool the solution to room temperature. Add, all at once, 10 ml of saturated HgCl_2 solution, stir, again wash down the inside of the beaker, and allow the solution to stand for 2 to 3 min. Add 20 ml of H_2SO_4 - H_3PO_4 mixture if $\text{K}_2\text{Cr}_2\text{O}_7$ solution is the titrant or 20 ml of sulfuric-phosphoric-manganous sulfate mixture if KMnO_4 solution is the titrant, and dilute to 200 ml with water. Titrate the iron with either 0.05 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution, adding 2 to 3 drops of diphenylamine sulfonate indicator. The end point is reached when color persists for at least 20 s.

6.2.3.2 *Alternative Method for Determination of Iron by Atomic Absorption*—Dilute the filtrate obtained in 6.2.1.1 to 200 ml with water. Transfer a 50-ml aliquot of this solution to a 100-ml volumetric flask and add 10 ml of lanthanum solution (5.12). (Weight of sample may be varied to eliminate aliquot.) Dilute to volume with water. Prepare a standard by combining 10 ml of 1000 ppm iron solution (5.11) and 10 ml of lanthanum solution in a 100-ml volumetric flask and diluting to volume with water. Prepare a blank solution by combining 30 ml of HNO_3 (1 + 7) and 10 ml of lanthanum solution in a 100-ml volumetric flask and diluting to volume with water. Determine absorbances of the blank solution, the standard, and the sample at 372 nm using an air-acetylene flame.

NOTE 4—The wavelength of 372 nm is chosen for the determination to provide a less sensitive line and wider linear range for the iron absorbance. Linearity in this range of iron concentration should be checked for each individual instrumental setup and appropriate dilutions made if necessary. Alternatively, the 248.3-nm line may be used.

NOTE 5—If concentration mode is used and unknown is bracketed, a blank is unnecessary.

6.3 *Iron Blank*—Make blank determinations following the sample procedure and using the same amounts of all reagents for pyritic iron (6.2) and nonpyritic iron (6.1.5) when a separate sample is used for the pyritic iron determination.

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7. Calculation

7.1 *Sulfate Sulfur*—Calculate the percentage of sulfate sulfur as follows:

$$\text{Sulfate sulfur, \%} = [(A - B) \times 13.735]/W$$

where:

- A = grams of BaSO₄ precipitated,
- B = grams of BaSO₄ in the blank, and
- W = grams of sample used.

7.2 *Pyritic Sulfur—Titration of Iron Using KMnO₄ or K₂Cr₂O₇*:

7.2.1 Calculate the percentage of sulfur when the determination is made on the residue from the HCl extract as follows:

$$\text{Pyritic sulfur, \%} = [(A - B)N \times 6.412]/W$$

where:

- A = millilitres of titrant required for titration of iron in HNO₃ extract,
- B = millilitres of titrant required for titration of the blank,
- N = normality of the titrant expressed to four decimal places,
- W = grams of original coal sample.

7.2.1.1 Calculate the percentage of sulfur when the iron determination is made by atomic absorption as follows:

From absorbance:

$$\text{Pyritic sulfur, \%} = \frac{C - A}{B - A} \times \frac{4.58}{W}$$

where:

- A = absorbance of blank,
- B = absorbance of standard,
- C = absorbance of sample, and
- W = weight of sample extracted as selected from 6.1.1 or 6.2.1

If the values higher than 5 % pyritic sulfur are obtained, a new sample solution should be prepared from the 20 ml of the filtrate 6.2.1.1 and 10 ml of lanthanum solution, diluted to 100 ml the value obtained on this solution $\times$ 2.5 = percent pyritic sulfur.

From Concentrations—Instruments so designed can be set to read-out directly in percent pyritic sulfur by the following calculation:

$$\text{Pyritic sulfur, \%} = \frac{D \times V \times 1.14}{W \times 104}$$

where:

- D = concentration of iron in standard, ppm,
- V = volume of analytical solution as prepared in 6.2.3.2,

W = weight of sample represented by this solution.

For example, if a 2.0 g sample of coal is extracted, the filtrate diluted to 200 ml and a 50-ml aliquot diluted to 100 ml, the weight of coal represented in the 100 ml of analytical solution would be 0.5 g. The instrument's read-out for 100 ppm iron standard would then be:

$$\text{Pyritic sulfur, \%} = \frac{100 \times 100 \times 1.14}{0.5 \times 104} = 2.296$$

7.2.2 Calculate the percentage of sulfur when the determination is made on a separate sample as follows:

$$\text{Pyritic sulfur, \%} = \left(\frac{A - B_1}{W_1} - \frac{C - B_2}{W_2} \right) \times N \times 6.412$$

where:

- A = millilitres of titrant required for titration of iron in HNO₃ extract,
- B₁ = millilitres of titrant required for titration of the blank for HNO₃ extraction,
- C = millilitres of titrant required for titration of iron in HCl extract,
- B₂ = millilitres of titrant required for titration blank for HCl extraction,
- N = normality of the titrant,
- W₁ = grams of coal sample extracted with HNO₃, and
- W₂ = grams of coal sample extracted with HCl.

7.3 *Organic Sulfur*—The percentage of organic sulfur is obtained by subtracting the sum of the percentages of sulfate and pyritic sulfur from the percentage of total sulfur as determined by the Eschka method described in Methods D 3177.

8. Precision

8.1 *Repeatability*—Results of consecutive determinations carried out on the same sample in the same laboratory by the same operator using the same apparatus should not differ more than the following:

Sulfate sulfur	0.02
Pyritic sulfur, under 2 %	0.05
Pyritic sulfur, 2 % or more	0.10

8.2 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples

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7. Calculation

7.1 *Sulfate Sulfur*—Calculate the percentage of sulfate sulfur as follows:

$$\text{Sulfate sulfur, \%} = [(A - B) \times 13.735]/W$$

where:

A = grams of BaSO₄ precipitated,

B = grams of BaSO₄ in the blank, and

W = grams of sample used.

7.2 *Pyritic Sulfur—Titration of Iron Using KMnO₄ or K₂Cr₂O₇*:

7.2.1 Calculate the percentage of sulfur when the determination is made on the residue from the HCl extract as follows:

$$\text{Pyritic sulfur, \%} = [(A - B)N \times 6.412]/W$$

where:

A = millilitres of titrant required for titration of iron in HNO₃ extract,

B = millilitres of titrant required for titration of the blank,

N = normality of the titrant expressed to four decimal places,

W = grams of original coal sample.

7.2.1.1 Calculate the percentage of sulfur when the iron determination is made by atomic absorption as follows:

From absorbance:

$$\text{Pyritic sulfur, \%} = \frac{C - A}{B - A} \times \frac{4.58}{W}$$

where:

A = absorbance of blank,

B = absorbance of standard,

C = absorbance of sample, and

W = weight of sample extracted as selected from 6.1.1 or 6.2.1

If the values higher than 5 % pyritic sulfur are obtained, a new sample solution should be prepared from the 20 ml of the filtrate 6.2.1.1 and 10 ml of lanthanum solution, diluted to 100 ml the value obtained on this solution $\times 2.5$ = percent pyritic sulfur.

From Concentrations—Instruments so designed can be set to read-out directly in percent pyritic sulfur by the following calculation:

$$\text{Pyritic sulfur, \%} = \frac{D \times V \times 1.14}{W \times 104}$$

where:

D = concentration of iron in standard, ppm,

V = volume of analytical solution as prepared in 6.2.3.2,

W = weight of sample represented by this solution.

For example, if a 2.0 g sample of coal is extracted, the filtrate diluted to 200 ml and a 50-ml aliquot diluted to 100 ml, the weight of coal represented in the 100 ml of analytical solution would be 0.5 g. The instrument's read-out for 100 ppm iron standard would then be:

$$\text{Pyritic sulfur, \%} = \frac{100 \times 100 \times 1.14}{0.5 \times 104} = 2.296$$

7.2.2 Calculate the percentage of sulfur when the determination is made on a separate sample as follows:

$$\text{Pyritic sulfur, \%} = \left(\frac{A - B_1}{W_1} - \frac{C - B_2}{W_2} \right) \times N \times 6.412$$

where:

A = millilitres of titrant required for titration of iron in HNO₃ extract,

B₁ = millilitres of titrant required for titration of the blank for HNO₃ extraction,

C = millilitres of titrant required for titration of iron in HCl extract,

B₂ = millilitres of titrant required for titration blank for HCl extraction,

N = normality of the titrant,

W₁ = grams of coal sample extracted with HNO₃, and

W₂ = grams of coal sample extracted with HCl.

7.3 *Organic Sulfur*—The percentage of organic sulfur is obtained by subtracting the sum of the percentages of sulfate and pyritic sulfur from the percentage of total sulfur as determined by the Eschka method described in Methods D 3177.

8. Precision

8.1 *Repeatability*—Results of consecutive determinations carried out on the same sample in the same laboratory by the same operator using the same apparatus should not differ more than the following:

Sulfate sulfur	0.02
Pyritic sulfur, under 2 %	0.05
Pyritic sulfur, 2 % or more	0.10

8.2 *Reproducibility*—The means of results of duplicate determinations carried out by different laboratories on representative samples

A P P E N D I X B

SULPHUR ANALYSIS DATA AND CHANNEL
SAMPLE HISTOGRAMS

CHANNEL SAMPLE NO. I
LINGAN MINE BOTTOM 7E WALL
NOVEMBER 26, 1981

TOTAL INCREMENT					-24 MESH					+24 MESH				
Wt.	St	Sp	So	Wt.	Wt%	St	Sp	So	Wt.	Wt%	St	Sp	So	
A	1601	0.70	0.12	0.58	32	2.00	0.68	0.11	0.58	1569	98.00	0.70	0.12	0.58
B	2145	0.57	0.07	0.50	42	1.96	0.59	0.05	0.54	2103	98.04	0.57	0.07	0.50
C	1576	1.60	1.11	0.49	28	1.78	1.03	0.62	0.42	1548	98.22	1.61	1.12	0.49
D	2144	0.49	*	*	78	3.64	0.33	0	0.33	2066	96.36	0.50	*	*
E	1388	0.45	*	*	53	3.82	0.40	0.04	0.37	1335	96.18	0.45	*	*
F	1414	0.49	0.01	0.48	47	3.32	0.43	0.04	0.40	1367	96.68	0.49	0.01	0.48
G	1180	0.58	0.03	0.55	87	7.37	0.43	0	0.43	1093	92.63	0.59	0.03	0.56
H	1168	1.14	0.60	0.54	51	4.37	1.07	0.61	0.46	1117	95.63	1.14	0.60	0.54
I	1156	1.20	*	*	76	6.57	1.11	0.46	0.65	1080	93.43	1.21	*	*
J	1129	2.04	1.40	0.64	49	4.34	1.65	0.99	0.66	1080	95.66	2.06	1.43	0.63
K	1237	1.64	1.01	0.63	31	2.51	1.64	1.14	0.50	1206	97.49	1.64	1.01	0.63
L	1336	1.72	1.07	0.65	52	3.89	1.46	0.88	0.58	1284	96.11	1.73	1.08	0.65
M	1409	1.31	0.64	0.67	12	0.85	1.27	0.56	0.71	1397	99.15	1.31	0.64	0.67
N	1320	0.50	*	*	16	1.21	0.59	0.06	0.53	1304	98.79	0.50	*	*
O	1529	1.52	0.91	0.61	22	1.44	1.43	0.70	0.73	1507	98.56	1.52	0.91	0.61
	21732 (15724)	1.02	0.59	0.57	676	3.11	0.86	0.36	0.50	21056 (15271)	96.89	1.02	0.60	0.57

Note: Wt. percentages are of total increment
*Insufficient Sample

CHANNEL SAMPLE NO. I
SINK AND FLOAT

	1.29 S.G. FLOAT					1.35 S.G. FLOAT					1.35 S.G. SINK					
	Wt.	%	St	Sp	So	Wt.	%	St	Sp	So	Wt.	%	St	Sp	So	
A	1539	98.28	0.68	0.10	0.58	20	1.28	1.63	1.32	0.31	7	0.45	1.95	1.72	0.23	1566
B	2089	99.24	0.57	0.07	0.51	9	0.43	0.53	0.01	0.52	7	0.33	0.47	0.41	0.06	2105
C	1347	87.02	0.69	0.24	0.46	89	5.75	3.29	2.54	0.75	112	7.23	11.40	10.55	0.86	1548
D	1992	96.05	0.50	0.0	0.50	70	3.38	0.45	0.26	0.19	12	0.58	0.60	*	*	2074
E	1313	98.50	0.45	0.0	0.45	10	0.75	0.46	*	*	10	0.75	0.18	0.23	0.0	1333
F	1357	99.05	0.49	0.01	0.49	9	0.66	0.43	0.02	0.41	4	0.29	0.59	0.66	0.0	1370
G	1015	92.44	0.57	0.0	0.57	34	3.10	0.60	0.06	0.54	49	4.46	0.99	0.68	0.31	1098
H	992	88.49	0.88	0.34	0.55	88	7.85	3.02	2.47	0.55	41	3.66	3.39	2.71	0.68	1121
I	947	87.36	0.80	0.22	0.59	110	10.15	3.84	3.25	0.59	27	2.49	4.80	*	*	1084
J	889	82.09	1.15	0.52	0.63	83	7.66	4.49	3.81	0.68	111	10.25	7.54	6.89	0.65	1083
K	961	79.42	1.11	0.45	0.67	198	16.36	3.27	2.76	0.66	51	4.21	5.31	4.82	0.50	1210
L	943	73.56	0.95	0.34	0.62	244	19.03	3.33	2.72	0.61	95	7.41	5.40	4.28	1.12	1282
M	1179	89.93	1.10	0.49	0.61	79	6.03	3.28	2.61	0.68	53	4.04	3.03	2.15	0.89	1311
N	1303	99.54	0.50	0.06	0.44	4	0.31	0.46	*	*	2	0.15	1.21	*	*	1309
O	1220	81.46	0.94	0.32	0.63	210	13.91	3.73	3.02	0.72	70	4.64	5.10	4.93	0.17	1510
Tot.	19096	90.92	0.72	0.18	0.54	1257 (1243)	5.98	3.15	2.59	0.62	651 (610)	3.10	5.78	5.28		21004

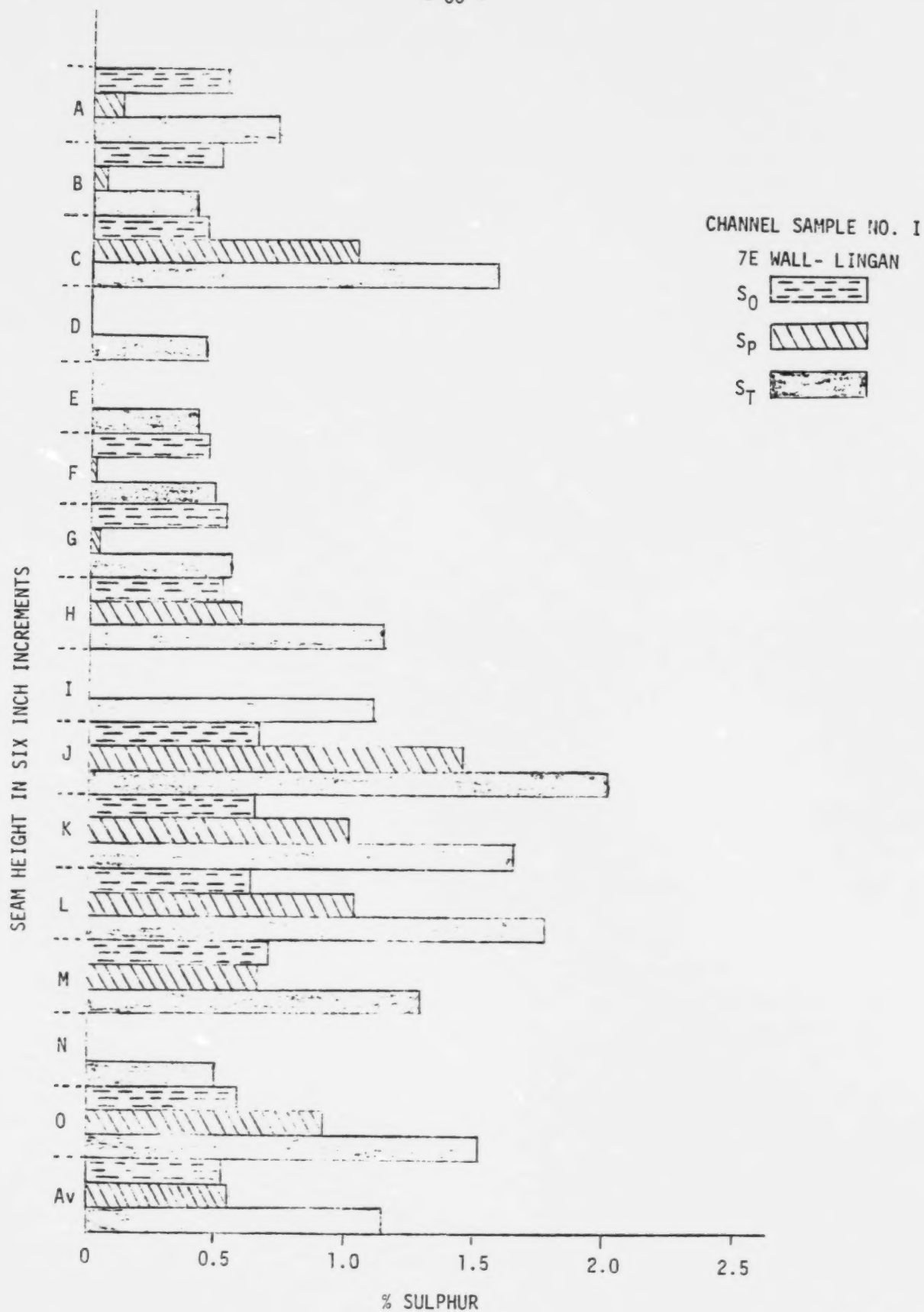
Note: All averages are weighted.

Sink Float percentages are of total sink-float weight

*Insufficient sample

CHANNEL SAMPLE NO. I
ASH IN S/F AND FINES

	1.29F	1.35F	1.35S	-24M	
A	1.24	4.02	55.96	5.27	A
B	1.59	5.25	32.80	2.71	B
C	1.37	5.77	19.45	5.10	C
D	1.36	4.40	9.96	4.48	D
E	1.55	8.62	18.37	4.75	E
F	1.11	5.69	22.62	5.28	F
G	1.18	4.83	13.27	5.50	G
H	1.40	5.27	23.97	9.82	H
I	1.44	5.50	28.05	6.99	I
J	1.81	6.27	17.22	8.65	J
K	1.43	4.70	27.53	11.71	K
L	1.53	4.56	17.22	7.85	L
M	1.80	5.37	11.98	5.36	M
N	1.61	5.88	16.75	3.54	N
O	1.53	5.68	58.89	12.02	C



CHANNEL SAMPLE NO. 2
LINGAN MINE - TOP 3 WEST WALL
DECEMBER 24, 1981

TOTAL INCREMENT			-24 MESH			+24 MESH		
Wt.	ST	Sp	So	Wt	Wt. %	St	Sp	So
A 1162	3.32	2.65	0.67	21	1.8	4.95	4.37	0.58
B 2995	2.17	1.77	0.40	96	3.2	3.35	2.86	0.49
C 1382	2.95	2.27	0.68	26	1.9	5.86	4.92	0.94
D 2718	1.91	1.52	0.39	60	2.5	2.57	2.01	0.56
E 2611	0.46	0.02	0.44	81	3.0	0.86	0.35	0.51
F 2259	0.50	0.06	0.44	171	7.5	0.61	0.42	0.19
G 2664	0.51	0.06	0.45	139	5.2	0.47	0.19	0.28
H 2426	0.78	0.29	0.49	47	1.9	0.79	0.26	0.53
I 2259	2.90	2.35	0.55	102	4.5	2.79	2.17	0.62
J 2052	0.81	0.22	0.59	68	3.3	0.91	0.47	0.44
K 2988	0.82	0.21	0.61	117	3.9	0.76	0.27	0.49
L 2300	2.77	2.19	0.58	98	4.3	2.64	2.10	0.54
M 3104	2.78	2.25	0.53	149	4.8	2.44	1.76	0.68
30920	1.64	1.14	0.50	1175	3.81	1.60	1.28	0.32
				29745	96.19	1.65	1.13	0.52

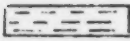
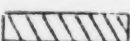
CHANNEL SAMPLE NO. 2
SINK AND FLOAT

1.30 S.G. FLOAT						1.35 S.G. FLOAT						1.35 S.G. SINK						S/F	
Wt.	%	St	Sp	So	Wt.	%	St	Sp	So	Wt.	%	St	Sp	So	Wt.				
A 150	13.1	1.94	1.03	0.91	325	28.5	2.46	2.00	0.46	666	58.4	4.03	3.26	0.77	1141	A			
B 1994	68.8	1.12	0.80	0.32	583	20.1	3.59	2.92	0.67	322	11.1	5.89	5.48	0.41	2899	B			
C 1055	77.8	1.85	0.15	0.70	158	11.7	5.08	4.23	0.85	143	10.5	8.34	7.92	0.42	1356	C			
D 2247	84.5	1.06	0.59	0.47	257	9.7	4.30	3.98	0.32	154	5.8	10.99	0.89	0.10	2658	D			
E 2509	99.2	0.44	0.00	0.44	12	0.5	0.98	0.56	0.42	9	0.3	2.99	2.72	0.27	2530	E			
F 1945	93.2	0.49	0.01	0.48	31	1.5	0.83	0.48	0.35	112	5.3	0.67	0.25	0.42	2088	F			
G 2327	92.1	0.50	0.01	0.49	85	3.4	0.71	0.39	0.32	113	4.5	0.81	0.72	0.09	2525	G			
H 1980	83.2	0.62	0.06	0.56	357	15.0	1.51	1.37	0.14	42	1.8	2.38	2.02	0.36	2379	H			
I 1475	68.4	1.40	0.75	0.65	201	9.3	4.03	3.58	0.45	481	22.3	7.14	6.82	0.32	2157	I			
J 1949	98.2	0.77	0.18	0.59	14	0.7	1.76	1.61	0.15	21	1.1	4.17	3.76	0.41	1984	J			
K 2799	97.5	0.81	0.19	0.62	23	0.8	2.13	1.63	0.50	49	1.7	1.21	0.87	0.33	2871	K			
L 795	36.1	1.95	1.01	0.94	712	32.3	2.50	1.84	0.66	695	31.6	4.10	3.42	0.68	2202	L			
M 1738	58.8	1.58	0.89	0.69	695	23.5	2.48	2.20	0.28	522	17.7	7.35	7.01	0.34	2955	M			
22963	77.20	0.93	0.38	0.55	3453	11.60	2.80	2.37	0.43	3329	11.20	5.41	4.92	0.49	29745				

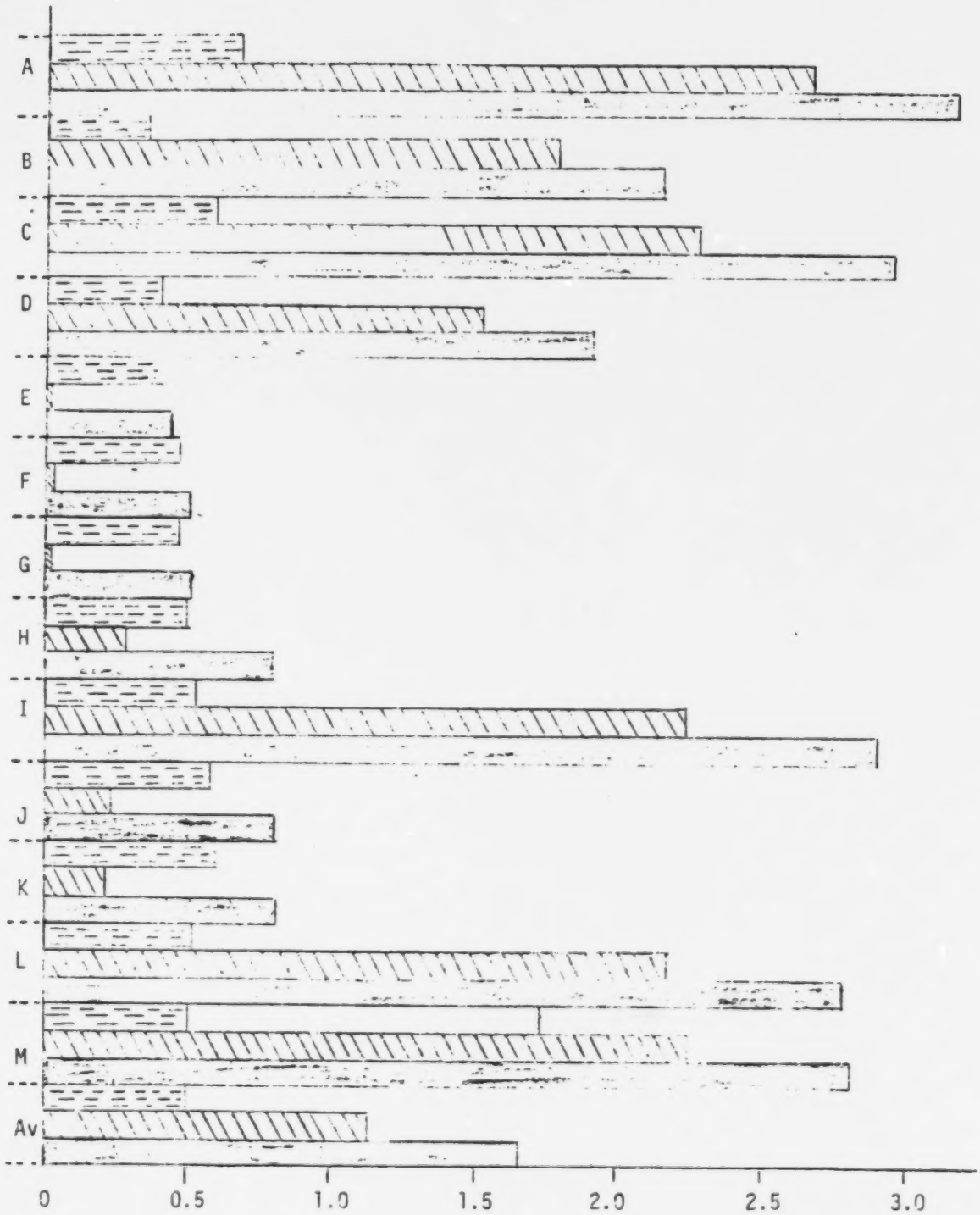
CHANNEL SAMPLE NO. 2
ASH IN S/F AND FINES

	1.30F	1.35F	1.35S	-24M	
A	4.07	6.75	15.16	20.57	A
B	1.69	6.28	50.53	22.60	B
C	2.03	6.28	13.51	23.48	C
D	1.73	6.50	16.66	10.74	D
E	1.23	5.49	28.08	6.50	E
F	1.04	5.75	43.24	11.55	F
G	1.15	6.62	33.48	10.10	G
H	1.84	7.88	17.27	5.56	H
I	2.17	5.84	10.88	7.89	I
J	1.04	4.49	14.64	5.98	J
K	1.02	4.89	16.16	4.96	K
L	2.83	7.29	11.78	10.00	L
M	2.88	5.40	12.80	7.25	M

Channel Sample No. 2
3 West Wall- Langan

 S₀
 S_p
 S_T

SEAM HEIGHT IN SIX INCH INCREMENTS



% SULPHUR

CHANNEL SAMPLE NO. 3
LINGAN MINE - TOP 7 EAST WALL
DECEMBER 23, 1981

TOTAL INCREMENT				-24 MESH						+24 MESH					
	Wt.	St.	Sp.	So.	Wt.	Wt. %	St.	Sp.	So.	Wt.	Wt. %	St.	Sp.	So.	
A	2618	4.36	3.29	1.07	49	1.87	4.44	4.17	0.27	2569	98.1	4.36	3.28	1.08	
B	1724	1.56	0.94	0.62	34	1.97	2.29	1.57	0.72	1690	98.0	1.55	0.93	0.69	
C	2595	3.31	2.47	0.84	45	1.73	3.21	2.10	1.11	2550	98.2	3.31	2.47	0.84	
D	2135	2.87	1.88	0.99	49	2.29	2.81	2.40	0.41	2086	97.7	2.88	1.87	1.01	
E	3107	2.47	1.79	0.68	71	2.28	2.91	2.44	0.47	3036	97.7	2.46	1.77	0.69	
F	3614	2.63	1.77	0.86	118	3.26	2.11	1.73	0.38	3496	96.7	2.65	1.77	0.88	
G	2745	2.58	1.69	0.89	142	5.17	1.78	1.40	0.38	2603	94.8	2.60	1.68	0.92	
H	2665	2.36	1.74	0.62	152	5.70	1.39	1.13	0.26	2513	94.3	2.42	1.78	0.64	
I	3700	1.20	0.69	0.51	121	3.27	1.10	0.63	0.47	3579	96.7	1.10	0.34	0.63	
J	2764	1.08	0.39	0.69	67	2.42	1.12	0.71	0.41	2697	97.5	1.02	0.37	0.65	
K	3484	1.19	0.61	0.58	80	2.29	1.09	0.62	0.47	3404	97.7	1.20	0.61	0.59	
L	3555	1.75	1.08	0.67	116	3.26	1.64	1.13	0.51	3439	96.7	1.76	1.08	0.68	
M	3312	2.55	1.93	0.62	73	2.20	2.14	1.60	0.54	3229	97.5	2.57	1.94	0.63	
	38018	2.23	1.52	0.71	1117	2.93	1.91	1.48	0.43	36891	97.0	2.27	1.53	0.74	

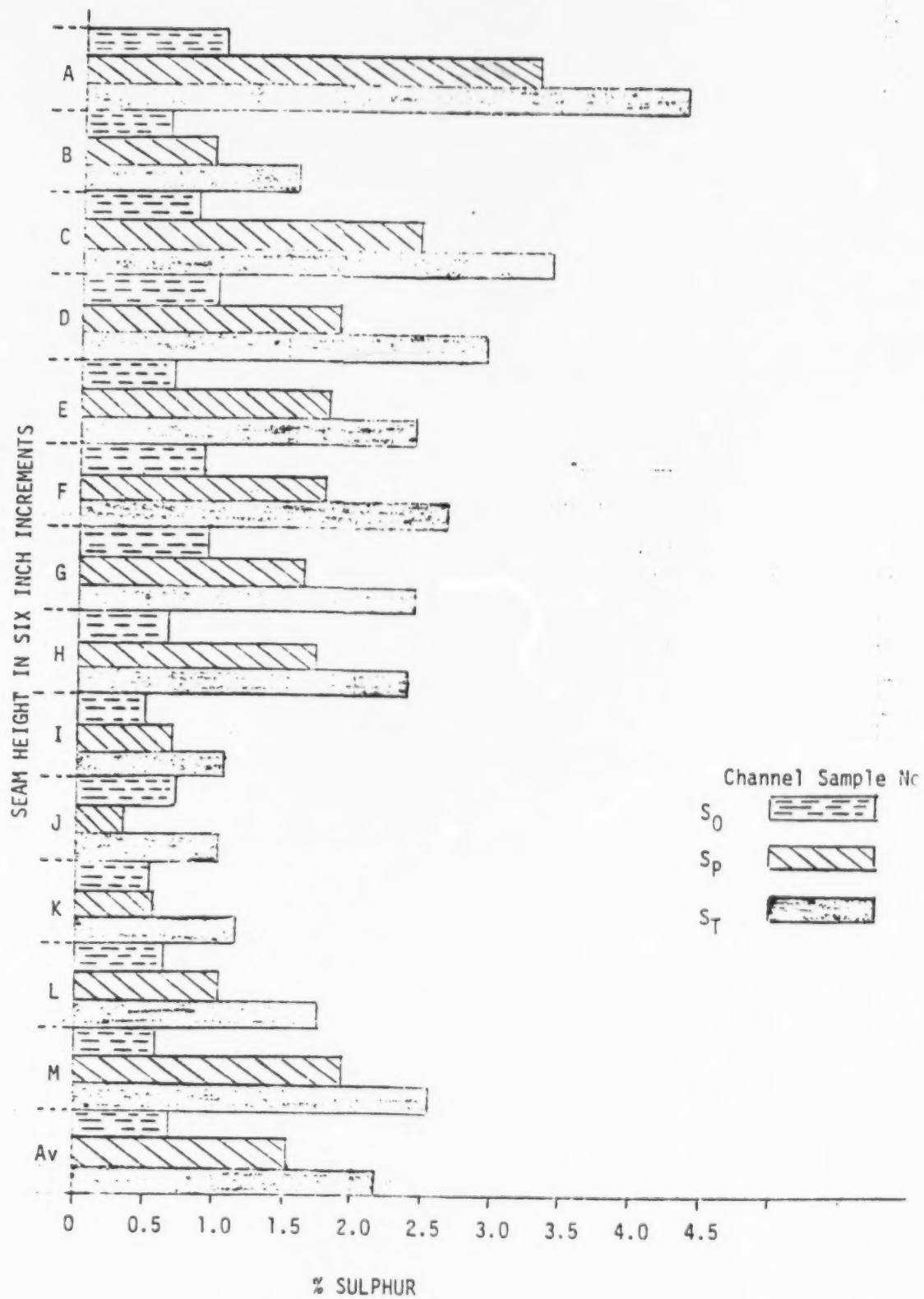
CHANNEL SAMPLE NO. 3

SINK AND FLOAT

1.30 S.G. - FLOAT										1.35 S.G. - SINK					S/F	
	Wt.	%	St.	Sp.	So.	Wt.	%	St.	Sp.	So.	Wt.	%	St.	Sp.	So.	Wt.
A	1199	46.6	2.76	1.74	1.02	1116	43.3	5.07	3.81	1.26	260	10.1	8.73	8.05	0.68	2575
B	1426	84.1	1.44	0.76	0.68	30	1.8	4.11	3.58	0.53	239	14.1	1.95	1.62	0.33	1695
C	1961	76.7	2.75	2.00	1.75	468	18.3	4.80	3.45	1.35	127	5.0	6.49	6.14	0.35	2556
D	2030	97.0	2.81	1.80	1.01	40	1.9	3.27	2.76	0.51	22	1.1	9.06	6.89	2.17	2092
E	2727	89.5	2.28	1.58	0.70	241	7.9	3.64	2.85	0.79	79	2.6	5.05	4.71	0.34	3047
F	3258	93.9	2.54	1.63	0.91	154	4.5	3.98	3.64	0.34	56	1.6	5.80	5.51	0.29	3468
G	2476	95.0	2.45	1.58	0.96	110	4.2	4.08	3.40	0.68	21	0.8	6.45	6.32	0.13	2607
H	1477	58.3	1.78	0.95	0.83	509	20.3	3.53	3.04	0.49	526	20.9	3.18	2.92	0.26	2512
I	2365	91.6	0.98	0.32	0.66	105	4.1	3.27	2.84	0.43	113	4.3	1.68	1.50	0.18	2583
J	2566	94.5	1.01	0.34	0.67	28	1.0	2.00	1.24	0.76	122	4.5	1.13	0.82	0.31	2716
K	3317	97.3	1.20	0.60	0.60	17	0.5	3.12	2.64	0.48	74	2.2	1.09	0.91	0.18	3408
L	2027	58.9	1.71	1.04	0.67	456	13.3	3.57	3.06	0.51	959	27.8	1.03	0.24	0.79	3442
M	2188	67.3	1.09	0.40	0.69	477	14.7	3.03	2.62	0.11	584	18.0	7.75	7.06	0.69	3249
	29017	84.19	1.87	1.11	0.76	3751	22.38	4.08	3.27	0.81	3182	18.38	3.83	3.28	0.55	35950

CHANNEL SAMPLE NO. 3
ASH IN S/F AND FINES

	1.30F	1.35F	1.35S	-24M	
A	3.64	8.19	16.43	10.78	A
B	1.72	7.81	20.15	6.80	B
C	3.23	6.73	14.57	7.03	C
D	3.76	7.39	16.40	7.04	D
E	3.23	5.29	17.10	7.54	E
F	2.72	6.52	22.58	8.86	F
G	2.84	5.97	17.88	5.85	G
H	2.86	6.07	16.59	5.48	H
I	1.34	6.38	24.01	5.51	I
J	1.26	6.27	21.46	9.46	J
K	1.44	6.61	27.81	5.79	K
L	2.98	6.43	15.16	6.01	L
M	2.64	5.89	22.80	8.83	M



CHANNEL SAMPLE NO. 4
LINGAN MINE - 3 WEST WALL

TOTAL INCREMENT					-24 MESH					+24 MESH				
	Wt.	St.	Sp.	So.	Wt.	Wt. %	St.	Sp.	So.	Wt.	Wt. %	St.	Sp.	So.
A	2306	4.39	3.52	0.87	85	3.68	4.33	3.25	1.08	2221	96.32	4.40	3.54	0.86
B	2154	5.34	4.29	1.05	50	2.32	6.29	5.15	1.14	2104	97.68	5.32	4.27	1.05
C	1956	4.57	3.90	0.67	64	3.27	7.32	5.51	1.81	1892	96.73	4.48	3.85	0.63
D	1927	4.00	2.66	1.34	59	3.06	4.22	2.98	1.24	1868	96.94	4.00	2.65	1.35
E	1193	2.95	1.82	1.13	31	2.59	2.69	1.84	0.85	1162	97.41	2.96	1.82	1.14
F	2058	2.70	1.85	0.85	97	4.71	2.70	1.74	0.96	1961	95.29	2.71	1.86	0.85
G	1549	2.41	1.70	0.71	74	4.77	2.10	1.48	0.62	1475	95.23	2.43	1.72	0.71
H	2034	3.49	2.75	0.74	120	5.90	3.31	2.58	0.73	1914	94.10	3.51	2.77	0.74
I	1674	2.28	1.49	0.79	48	2.86	3.78	3.03	0.75	1626	97.14	2.24	1.45	0.79
J	1523	1.74	0.98	0.76	57	3.74	2.30	1.74	0.56	1466	96.26	1.72	0.96	0.76
K	2357	2.13	2.12	0.01	108	4.58	2.25	1.58	0.67	2249	95.42	2.89	2.18	0.71
L	2810	2.12	1.44	0.68	103	3.66	1.80	1.11	0.69	2707	96.34	2.13	1.46	0.67
M	2331	2.63	1.79	0.84	88	3.77	2.21	1.54	0.67	2243	96.23	2.65	1.80	0.85
	25872	3.23	2.38	0.85	984	3.80	3.28	2.40	0.88	24888	96.20	3.23	2.38	0.85

CHANNEL SAMPLE NO. 4

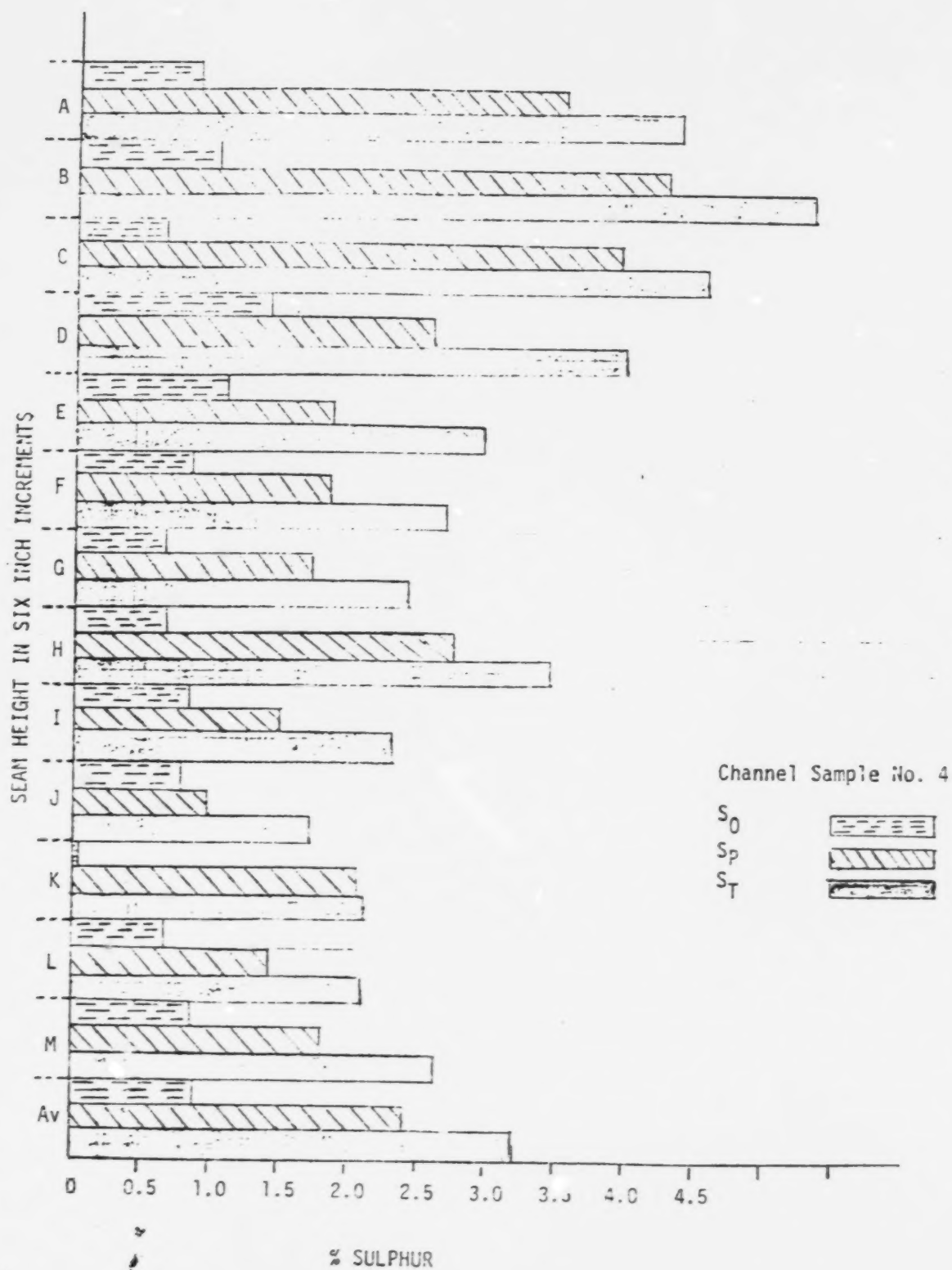
SINK AND FLOAT

1.30 S.G. - FLOAT						1.35 S.G. - FLOAT						1.35 S.G. - SINK						S/F	
	Wt.	%	St.	Sp.	So.	Wt.	%	St.	Sp.	So.	Wt.	%	St.	Sp.	So.	Wt.			
A	1325	59.79	2.13	1.15	0.98	495	22.33	4.43	3.97	0.46	396	17.87	12.0	11.03	0.97	2216	A		
B	925	44.02	2.46	1.19	1.27	804	38.26	5.17	4.72	0.45	372	17.70	12.8	10.99	1.81	2101	B		
C	1323	70.22	2.78	1.94	0.84	336	17.83	4.85	4.82	0.03	225	11.94	14.0	13.65	0.35	1884	C		
D	1225	65.57	3.31	2.03	1.28	612	32.76	5.01	3.59	1.42	31	1.66	11.7	9.07	2.63	1868	D		
E	1048	89.95	2.78	1.64	1.14	106	9.09	3.95	2.98	0.97	11	0.94	10.7	7.85	2.85	1165	E		
F	1850	92.92	2.53	1.71	0.82	115	5.77	4.07	3.09	0.98	26	1.30	10.0	7.72	2.28	1991	F		
G	1305	88.71	2.26	1.55	0.71	91	6.18	3.18	2.41	0.77	75	5.09	4.65	3.76	0.89	1471	G		
H	1084	56.93	2.09	1.25	0.84	305	16.01	4.00	3.10	0.90	515	27.04	6.21	5.80	0.41	1904	H		
I	1377	84.37	1.36	0.67	0.69	183	11.21	4.17	3.01	1.16	72	4.41	14.3	12.48	1.82	1632	I		
J	1262	86.02	1.33	0.58	0.78	175	11.93	3.52	2.68	0.84	30	2.04	7.96	6.94	1.02	1467	J		
K	1358	60.43	1.45	0.81	0.64	634	28.21	3.38	2.63	0.75	255	11.34	9.34	8.36	0.98	2247	K		
L	1840	67.99	1.39	0.76	0.63	663	24.50	3.19	2.52	0.67	203	7.50	5.39	4.34	1.05	2706	L		
M	1569	69.88	1.40	0.63	0.77	201	8.95	2.69	1.95	0.74	475	21.16	6.77	5.63	1.14	2245	M		
	17491	70.25	2.05	1.20	0.85	4720	18.96	4.15	3.42	0.73	2686	10.79	9.27	8.24	1.03	24897			

CHANNEL SAMPLE NO. 4

ASH IN S/F AND FINES

	1.30F	1.35F	1.35S	-24M	
A	2.78	5.72	20.01	15.88	A
B	3.30	7.28	17.60	12.75	B
C	3.72	7.93	15.50	11.90	C
D	4.16	6.22	14.71	7.49	D
E	3.24	5.30	13.56	5.88	E
F	3.00	5.67	12.41	7.03	F
G	3.06	5.27	27.74	8.55	G
H	2.80	5.82	13.68	9.98	H
I	1.85	5.53	20.08	8.50	I
J	1.78	6.00	13.42	6.68	J
K	2.68	5.44	18.21	7.95	K
L	2.60	5.46	20.30	8.56	L
M	2.50	7.79	44.31	16.20	M



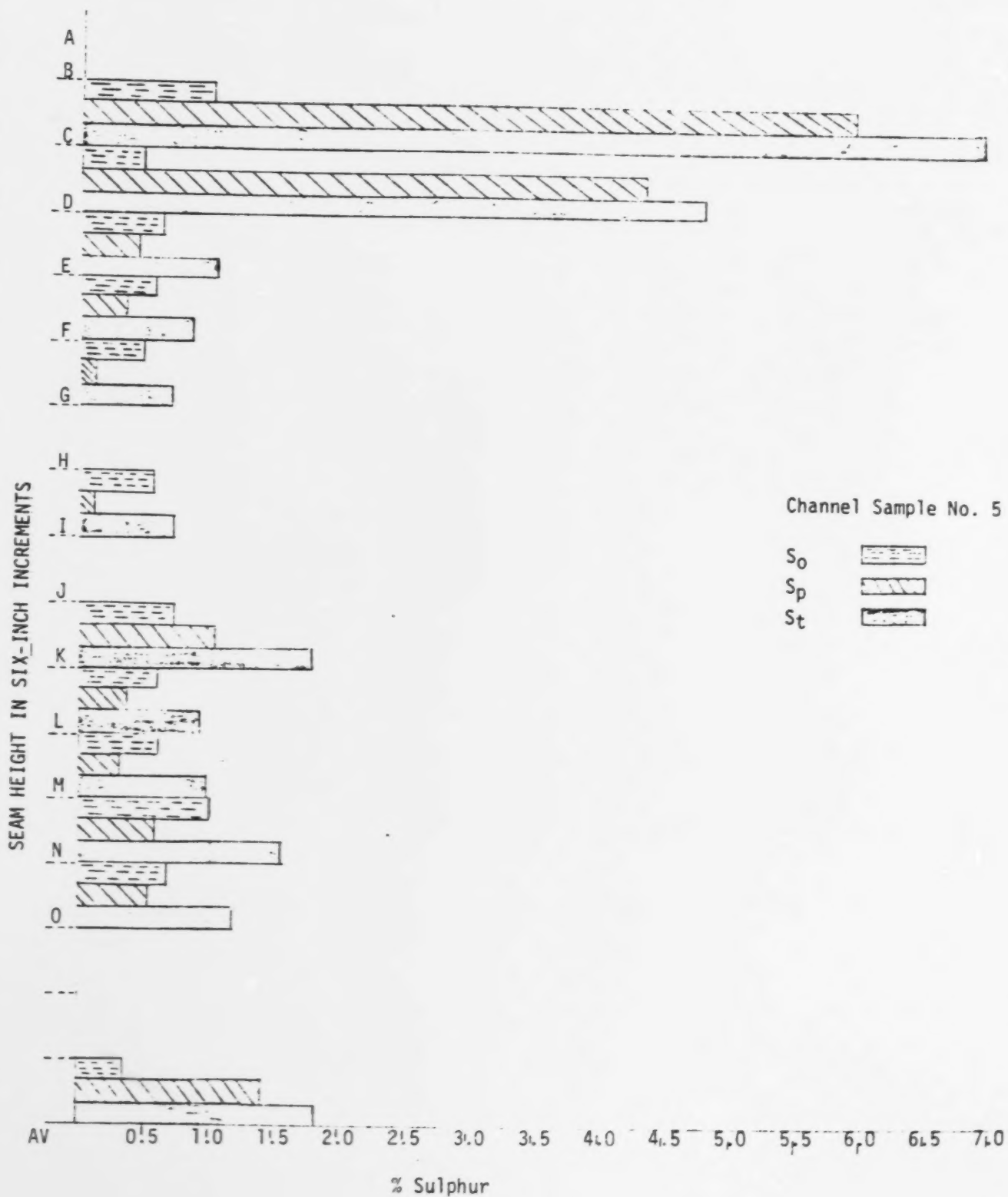
CHANNEL SAMPLE NO. 5
LINGAN MINE - 6 EAST WALL

Total Increment					-24 Mesh					+24 Mesh				
Wt.	ST	SP	SO		Wt.	Wt. %	ST	SP	SO	Wt.	Wt. %	ST	SP	SO
A	1000	6.91	3.91	1.00	54	5.4	3.78	2.87	0.91	946	94.6	7.15	6.19	0.96
B	1103	4.77	4.32	0.45	33	2.9	3.96	2.94	1.02	1070	97.1	4.80	4.36	0.44
C	1111	1.04	0.41	0.63	29	2.6	1.85	1.19	0.66	1082	97.4	1.02	0.39	0.63
D	891	0.83	0.29	0.54	23	2.6	0.98	0.41	0.57	868	97.4	0.83	0.28	0.55
E	856	0.64	0.17	0.47	38	4.4	0.81	0.31	0.50	818	93.6	0.63	0.16	0.47
F	600	0.73	*	*	29	4.8	0.91	0.38	0.53	571	93.2	0.72	*	*
G	794	0.71	0.17	0.54	51	6.4	0.70	0.26	0.44	743	93.6	0.67	0.17	0.50
H	773	0.54	*	*	45	5.8	0.71	0.19	0.52	728	94.2	0.53	*	*
I	870	1.76	1.05	0.71	51	5.8	1.35	0.72	0.63	819	94.2	1.79	1.07	0.72
J	864	0.89	0.35	0.54	54	6.2	0.86	0.70	0.16	810	93.8	0.90	0.32	0.58
K	824	0.94	0.28	0.66	29	3.5	1.01	0.41	0.60	795	96.5	0.93	0.28	0.65
L	829	1.54	0.55	0.99	29	3.5	1.75	0.97	0.78	800	96.5	1.53	0.53	1.00
M	938	1.17	0.51	0.66	45	4.8	1.35	0.60	0.75	893	95.2	1.16	0.50	0.66
N	585	1.04	*	*	17	2.9	1.17	0.54	0.63	568	97.1	1.04	*	*
O	831	1.02	*	*	37	4.4	0.96	0.34	0.62	794	95.6	1.02	*	*
12869	1.79	1.44	0.35		564	4.38	1.56	0.89	0.67	12305	96.62	1.80	1.42	0.38

CHANNEL SAMPLE NO. 5
SINK AND FLOAT

	1.30 S.G. Float					1.35 S.G. Float					1.35 S.G. Sink				
	Wt.	Wt. %	ST	SP	SO	Wt.	Wt. %	ST	SP	SO	Wt.	Wt. %	ST	SP	SO
A	262	27.6	2.53	1.53	1.00	141	14.9	4.91	4.08	0.83	546	57.5	9.91	8.94	0.97
B	302	28.2	1.72	1.05	0.67	331	30.9	3.97	3.19	0.78	437	40.9	7.57	7.55	0.02
C	1050	97.2	0.84	0.30	0.54	12	1.1	4.69	3.65	1.04	18	1.7	4.80	3.76	1.04
D	823	94.6	0.59	0.10	0.49	42	4.8	5.06	3.51	1.55	5	0.6	5.86	4.31	1.55
E	728	89.2	0.51	0.00	0.51	26	3.2	2.99	2.45	0.54	62	7.6	1.16	1.08	0.08
F	545	95.1	0.52	0.02	0.50	6	1.1	3.06	*	*	22	3.8	5.14	3.76	1.38
G	526	71.0	0.52	0.03	0.49	84	11.3	0.92	0.40	0.52	131	17.7	1.13	0.60	0.53
H	672	92.5	0.54	0.02	0.52	38	5.2	0.56	0.06	0.50	17	2.3	0.41	*	*
I	532	64.9	0.87	0.23	0.64	113	13.8	3.91	2.77	1.14	175	21.3	3.22	2.54	0.68
J	794	98.0	0.89	0.31	0.58	9	1.1	1.44	0.72	0.72	7	0.9	1.78	1.74	0.04
K	756	95.1	0.89	0.23	0.66	30	3.8	2.11	1.44	0.67	9	1.1	1.14	0.63	0.51
L	619	77.3	1.05	0.36	0.69	81	10.1	2.87	2.22	0.65	101	12.6	3.44	2.27	1.17
M	840	94.2	1.14	0.48	0.68	33	3.7	1.83	1.13	0.70	19	2.1	1.39	0.72	0.67
N	500	89.3	1.00	0.33	0.67	58	10.4	1.55	0.80	0.75	2	0.3	1.30	*	*
O	779	98.1	1.02	0.38	0.64	12	1.5	1.58	0.66	0.92	3	0.4	1.15	*	*
	9728	79.1	0.88	0.26	0.62	1016	8.3	3.33	2.51	0.82	1554	12.6	6.52	6.00	0.52

*Insufficient Sample.



APPENDIX C

TOTAL IRON AND FREE IRON

CHANNEL SAMPLE NO I

TOTAL AND FREE IRON

INCREMENT	-24 MESH		1.30 FLOAT		1.35 FLOAT		1.35 SINK	
	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON
A	0.62	0.52	0.20	0.11	1.59	0.43	2.93	1.43
B	0.28	0.24	0.13	0.07	0.43	0.42	3.97	3.61
C	0.95	0.41	0.35	0.14	2.45	0.14	9.59	0.37
D	--	0.96	0.27	0.28	1.06	0.83	2.67	1.77
E	1.13	1.09	0.41	0.41	--	--	6.07	5.87
F	0.70	0.66	0.22	0.21	1.22	1.20	6.09	5.51
G	--	0.60	0.27	0.27	1.14	1.09	1.93	1.34
H	1.15	0.45	0.38	0.08	2.40	0.23	3.56	1.18
I	0.84	0.43	0.35	0.16	3.15	0.29	5.25	3.19
J	1.20	0.33	0.55	0.09	0.18	0.17	6.46	0.37
K	1.31	0.31	0.49	0.09	2.41	--	4.83	0.60
L	1.08	0.31	0.37	0.07	2.48	0.10	4.19	0.43
M	1.06	0.57	0.54	0.11	2.52	0.23	2.87	0.98
N	0.61	0.56	0.40	0.34	--	--	--	--
O	1.31	0.70	0.43	0.15	2.73	0.08	5.44	1.11

CHANNEL SAMPLE NO. 2

TOTAL AND FREE IRON

INCREMENT	-24 MESH		1.30 FLOAT		1.35 FLOAT		1.35 SINK	
	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON
A	4.14	0.31	1.05	0.14	1.83	0.08	3.07	0.21
B	3.33	0.82	0.79	0.09	2.63	0.07	6.46	1.65
C	4.56	0.24	1.06	0.05	3.79	0.08	7.16	0.21
D	1.96	0.20	0.71	0.19	3.55	0.06	9.95	0.21
E	0.81	0.50	0.20	0.20	1.43	0.94	3.80	1.41
F	2.98	2.61	0.19	0.18	2.11	1.69	14.53	13.70
G	2.58	2.41	0.30	0.29	1.77	1.43	16.94	15.76
H	0.78	0.55	0.34	0.29	1.55	0.35	4.02	2.25
I	2.32	0.42	0.86	0.20	3.21	0.07	6.76	0.78
J	0.68	0.27	0.22	0.06	1.81	0.40	5.17	1.58
K	0.49	0.25	0.21	0.04	1.63	0.20	2.03	1.27
L	2.05	0.21	0.95	0.06	1.68	0.07	3.20	0.20
M	1.69	0.15	0.82	0.04	1.97	0.04	6.26	0.11

CHANNEL SAMPLE NO. 3
TOTAL AND FREE IRON

INCREMENT	-24 MESH		1.30 FLOAT		1.35 FLOAT		1.35 SINK	
	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON
A	3.90	0.24	1.57	0.04	3.52	0.18	7.27	0.21
B	1.51	0.13	0.72	0.04	3.42	0.26	2.29	0.87
C	1.98	0.14	1.79	0.03	3.09	0.06	6.12	0.73
D	2.32	0.21	1.63	0.05	3.27	0.85	6.99	0.95
E	2.21	0.07	1.42	0.03	2.54	0.04	4.34	0.21
F	1.62	0.10	1.47	0.04	3.35	0.16	5.53	0.28
G	1.30	0.07	1.41	0.02	3.07	0.09	5.87	0.33
H	0.86	0.07	0.85	0.02	2.75	0.08	2.64	0.08
I	0.85	0.30	0.33	0.05	2.63	0.14	2.24	0.92
J	1.35	0.73	0.36	0.06	1.51	0.42	5.14	4.36
K	0.74	0.20	0.58	0.05	2.50	0.08	5.05	4.34
L	1.10	0.11	0.96	0.05	2.84	0.16	1.76	1.55
M	1.54	0.13	0.37	0.02	2.38	0.08	6.50	0.31

CHANNEL SAMPLE NO. 4
TOTAL AND FREE IRON

INCREMENT	-24 MESH		1.30 FLOAT		1.35 FLOAT		1.35 SINK	
	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON
A	3.01	0.16	1.07	0.06	3.53	0.05	9.93	0.25
B	4.64	0.12	1.17	0.13	4.19	0.05	9.91	0.27
C	4.93	0.10	1.74	0.04	4.27	0.04	12.10	0.13
D	2.69	0.08	1.83	0.05	3.19	0.04	8.14	0.18
E	1.71	0.09	1.48	0.04	2.64	0.03	7.09	0.20
F	1.61	0.08	1.55	0.05	2.74	0.03	6.95	0.18
G	1.38	0.09	1.40	0.04	2.15	0.04	3.83	0.53
H	2.36	0.10	1.15	0.05	2.77	0.05	5.21	0.12
I	2.86	0.20	0.62	0.03	2.71	0.07	11.46	0.51
J	1.68	0.15	0.55	0.04	2.43	0.08	6.34	0.25
K	1.46	0.08	0.72	0.01	2.36	0.05	7.53	0.20
L	1.04	0.07	0.71	0.04	2.25	0.04	4.05	0.24
M	1.56	0.21	0.60	0.05	1.77	0.06	5.62	0.68

CHANNEL SAMPLE NO. 5
TOTAL AND FREE IRON

INCREMENT	-24 MESH		1.30 FLOAT		1.35 FLOAT		1.35 SINK	
	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON	TOTAL IRON	FREE IRON
A	3.57	1.05	1.46	0.12	3.66	0.08	8.29	0.45
B	3.18	0.60	0.99	0.07	2.92	0.12	7.19	0.57
C	1.48	0.44	0.38	0.12	3.33	0.13	4.29	0.99
D	0.85	0.49	0.25	0.16	3.25	0.17	4.50	0.72
E	0.83	0.56	0.18	0.18	2.78	0.63	3.89	2.94
F	0.92	0.59	0.29	0.27	--	--	4.30	1.00
G	2.16	1.93	0.65	0.62	2.82	2.47	11.62	11.09
H	0.77	0.60	0.32	0.30	0.28	2.23	--	--
I	0.95	0.32	0.43	0.23	2.78	0.35	3.26	1.03
J	1.29	0.68	0.42	0.15	1.42	0.79	5.04	3.49
K	0.93	0.57	0.53	0.33	2.92	1.66	7.12	6.57
L	1.15	0.30	0.45	0.13	2.27	0.32	2.67	0.68
M	0.67	0.14	0.53	0.12	1.27	0.28	1.12	0.49
N	0.65	0.18	0.34	0.05	0.85	0.15	--	--
O	0.51	0.21	0.37	0.04	0.64	0.06	--	--

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